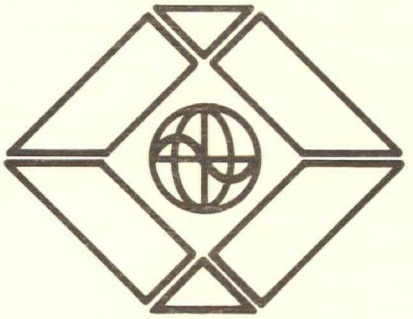
REPORT

EMISSION MEASUREMENTS AT THE ASARCO LEAD SMELTER IN EAST HELENA, MONTANA

II. RESULTS, CALCULATIONS AND APPENDIXES

Solutions for energy, environment & technology



PACIFIC ENVIRONMENTAL SERVICES, INC.

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EMISSION MEASUREMENTS AT THE ASARCO LEAD SMELTER IN EAST HELENA, MONTANA

II. RESULTS, CALCULATIONS AND APPENDIXES

May 1980

EPA Contract 68-01-4140

Task Order 60

Performed for

EPA Region VIII
301 South Park
Helena, Montana 59601

John Busik, Project Officer
Thomas O. Harris, Task Manager

Pacific Environmental Services, INC.

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1.0 INTRODUCTION

The emissions from the ASARCO smelter in East Helena, Montana, had never been evaluated as to compliance with the state of Montana's State Implementation Plan as promulgated in the Federal Register at 40 FR 10877, May 31, 1972. It was important that this be done, and Pacific Environmental Services (PES) was assigned this project under contract 68-01-4140, Task number 60. The testing and analyses were carried out by a sub-contractor, Certified Testing Laboratories (CTL) of South Gate, California, with some additional manpower supplied from PES.

The ASARCO plant is a custom smelter serving numerous small mining operations and handling a variety of ores. The plant processes an average of 300,000 tons of material per year, producing lead, copper, gold, silver, and zinc. A schematic (from ASARCO) of the major plant components is shown in Figure 1-1. The sampling locations are identifiable on the figure as follows:

1. Sinter plant main stack flue
2. Flue to the zinc fuming plant stack
3. Blast furnace baghouse; three stacks

There was also a tentative plan to sample the kettle ventilation baghouse stack, not shown on the figure, but this was not carried out.

The sinter plant main stack was readily accessible from existing platforms and was already ported for sampling. The flue to the zinc stack required additional ports for a vertical traverse, which were put in by ASARCO. Access to this stack (a large horizontal stack) required erection of scaffolding, which was done while the main stack was being sampled.

The blast furnace baghouse stacks are square wooden stacks about eleven feet across, connecting at right angles with the interior of the building just at the three gables on one end. The sample ports,

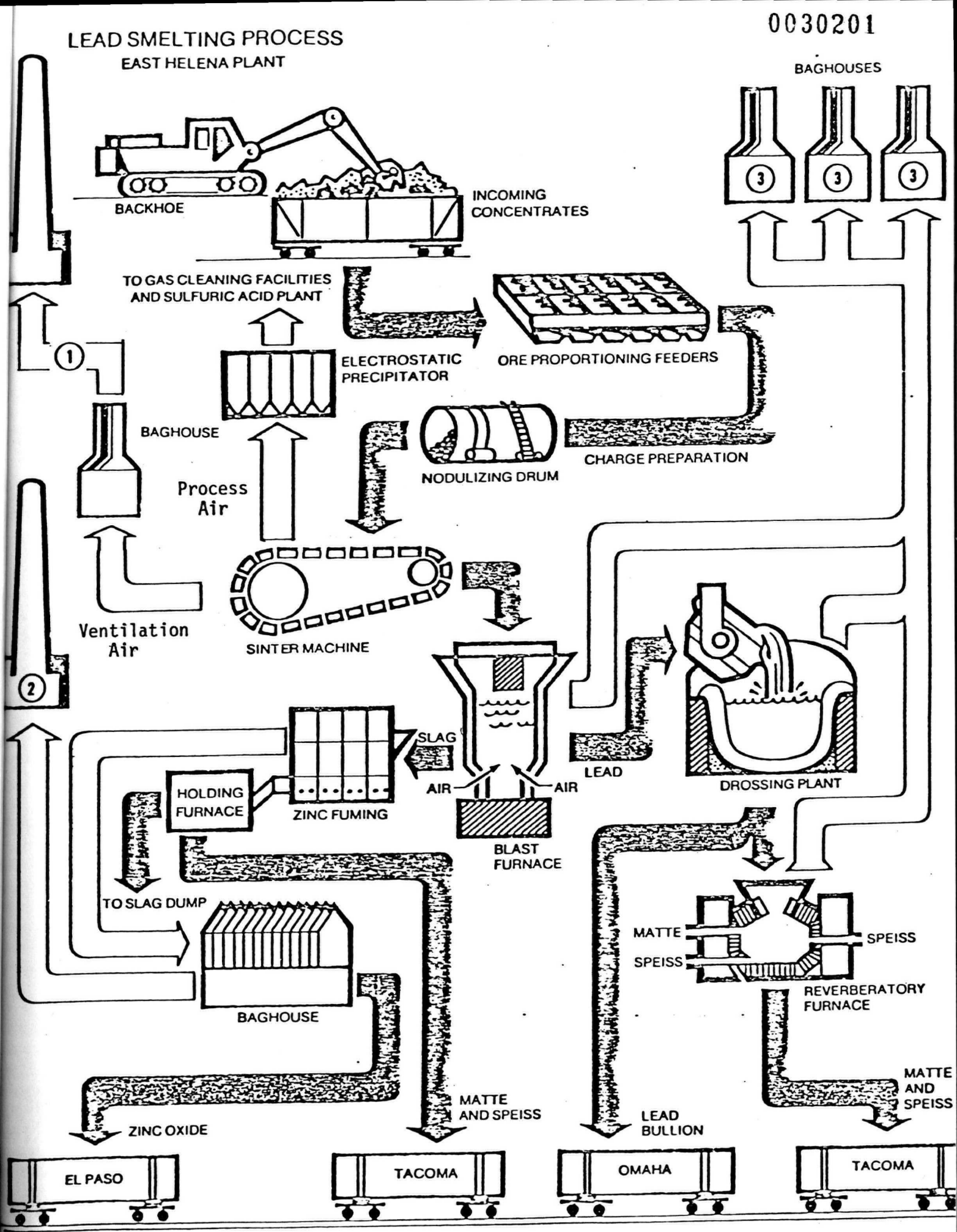

Figure 1-1. A Schematic of Major Plant Components

therefore, had to be a minimum of 22 feet above the right angle. Since the roof top was roughly 80 feet off the ground, it was only practical to sample from the roof side of the stacks. ASARCO constructed three wooden pads across the roof peaks next to the stacks, to serve as the support for scaffolding needed to reach the sampling location 22 feet higher. The scaffolding was hoisted up with a davit to walkways just below the roof and manhandled the rest of the way to the pads. This scaffolding was also erected while the main stack was tested. The pad at baghouse stack number one was slightly under size, so that two 5' x 7' scaffolds did not fit crosswise, as intended. Instead, a single scaffold was placed lengthwise. (By oversight, the task manager was not informed of the undersized pad in time to have it altered. The single scaffold made the two sample ports outboard of the scaffold difficult of access.) All scaffolds were strapped securely to the stacks.

2.0 SAMPLING AND ANALYSES

2.1 SAMPLING

Sampling work began on October 18, 1979 and was continued through to October 27 without breaks. The main stack and the zinc stack were each tested three times, requiring two days each. The plan was to sample the three baghouse stacks simultaneously with three trains, to which end two additional 11 foot probes had been acquired (to reach across the stack from one side). One balky sampling train delayed actual testing to the point where, with concurrence of the EPA observers, the stacks were instead tested three times each, in pairs. Test procedures are described in the Appendix.

2.2 ANALYSES

Filter samples were returned to CTL for reweighing, extraction, and analysis by atomic absorption spectroscopy for lead, arsenic, and cadmium. The analytical procedures are described in the Appendix. The impinger catches and probe washes were combined for determination of dry residue weights and the same three metals. The lead and cadmium were run by standard flame atomic absorption methods, the arsenic after generation of the hydride (arsine). Each extract was run in triplicate. Each of the three determinations was made by a different technician with his own reference standards. One of the three also ran standard additions to all samples to demonstrate lack of matrix effects. All lab determinations were averaged.

2.3 SAMPLE CUSTODY

All samples (filters, washings, impinger catches) from point of collection were kept in the custody of the testing sub-contractor and brought back by him to his laboratory for analysis.

After sample extraction and analyses, the residues were stored and at this date remain in his custody (Certified Testing Laboratories, South Gate, California). According to prior ASARCO-EPA agreement, and upon verbal approval from Mike Davenport of the EPA office in Helena, portions of these extracts were sent to ASARCO.

3.0 SUMMARY OF RESULTS

All sampling rates were within the acceptable range for percent isokinetic (actual range 93-101%). A summary of results is given in Table 3-1, followed by a more detailed tabulation of data and calculated values in Table 3-2. (Copies of the field work sheets are in the Appendix.)

Total particulates combined on the filter and in combined impinger and washings in three runs were: main stack - 0.0491 to 0.0626 gr/DSCF; flue to the zinc stack - 0.0116 to 0.0137 gr/DSCF; and baghouse stacks - 0.00693 to 0.0170 gr/DSCF. The major part of the total, except in a few baghouse samples, was in the impingers and washings.

The lead emissions in kg/day were: main stack - 32.0 to 49.6; flue to the zinc stack - 4.98 to 16.5; baghouse stacks - 6.65 to 12.9. Cadmium emissions in kg/day were: main stack - 0.667 to 0.789; flue to the zinc stack - 0.098 to 0.341; baghouse stacks - 1.13 to 3.52. Arsenic emissions in kg/day were: main stack - 28.2 to 49.1; flue to the zinc stack - 2.28 to 8.03; baghouse stacks - 0.831 to 2.66. Thus lead emissions were highest in the main stack and fairly similar in the flue to the zinc and baghouse stacks. Cadmium was highest in baghouse stacks and lowest in the flue to the zinc stacks. Main stack arsenic emissions were about as high as those of lead.

Table 3-1. SUMMARY OF TEST RESULTS

Location	Run	Sample Volume DSCF	Particulate Concentration gr/DSCF			Mass Emission Rate kg/day		
			Filter	Impinger & Washes	Total	Lead	Cadmium	Arsenic
Main Stack	1	65.2	0.00679	0.0423	0.0491	33.2	0.741	28.2
	2	46.4	0.00910	0.0535	0.0626	49.6	0.789	49.1
	3	44.1	0.00738	0.0536	0.0609	32.0	0.667	48.8
Flue to Zinc Stack	1	41.2	0.00232	0.00986	0.0122	16.5	0.271	3.87
	2	44.1	0.00165	0.0121	0.0137	5.45	0.098	2.28
	3	43.7	0.00170	0.00989	0.0116	4.98	0.341	8.03
Baghouse Stack #1	3	83.7	0.00354	0.00706	0.0106	9.06	2.80	1.00
	4	74.7	0.00391	0.00922	0.0131	9.13	2.44	2.14
	5	87.6	0.00267	0.00569	0.00836	7.14	3.52	1.71
Baghouse Stack #2	1*	76.5	0.00508	0.00801	0.0131	12.9	3.46	0.831
	2	76.6	0.00431	0.00417	0.00848	10.8	1.72	1.74
	3	77.3	0.00469	0.00521	0.00990	8.05	2.30	1.07
	4	83.2	0.00549	0.0105	0.0160	10.6	3.18	2.66
Baghouse Stack #3	1	65.2	0.00436	0.0126	0.0170	10.8	2.52	1.58
	2	67.3	0.00346	0.00489	0.00835	7.46	1.13	1.52
	5	72.8	0.00318	0.00375	0.00693	6.65	3.17	1.00

* Run not valid due to leak from broken glass filter.
(Break occurred after end of run)

Table 3-2. TEST DATA SUMMARY

CTL - ENVIRONMENTAL SERVICES
2905 E. Century Boulevard
South Gate, CA. 90280

Report to: Pacific Environmental Services
2716 Oceanpark Blvd., Suite 3010
Santa Monica, California 90405

Source Test Work
ASARCO Smelter
Helena, Montana

LOCATION	RUN #	and DATE	METERED VOLUME	BAROMETRIC PRESSURE (Inches Hg)	AVERAGE ΔH (Inches H_2O)	AVERAGE METER TEMP. °F	METER CORRECTION FACTOR	VOLUME GAS SAMPLED AT STANDARD CONDITIONS 68°F 29.92 Inches Hg (cubic feet)
Main Stack	1	9/21/79	74.3	26.0	2.5	69.0	1.00	65.2
	2	9/21/79	54.4	26.0	2.5	84.6	1.00	46.4
	3	9/21/79	53.4	26.0	2.3	103	1.00	44.1
Flue to Zinc Stack	1	9-22-79	49.1	25.9	0.98	89.0	1.00	41.2
	2	9-23-79	49.6	26.0	1.08	60.8	1.00	44.1
	3	9-23-79	50.5	26.0	1.13	73.8	1.00	43.7
Bag House Stack #1	3	9-28-79	98.6	25.9	3.80	82.4	0.996	83.7
	4	9-28-79	89.4	25.9	3.09	90.8	0.996	74.7
	5	9-29-79	104.4	26.1	3.98	92.4	0.996	87.6
Bag House Stack #2	1	*9-26-79	89.0	25.9	3.06	79.1	1.00	76.5
	2	9-27-79	90.7	26.0	3.05	90.1	1.00	76.6
	3	9-28-79	88.2	25.9	3.07	68.1	1.00	77.3
	4	9-28-79	99.6	25.9	3.63	96.0	1.00	83.2
Bag House Stack #3	1	9-26-79	77.3	25.9	2.35	84.2	0.996	65.2
	2	9-27-79	80.4	26.0	2.42	90.0	0.996	67.3
	5	9-29-79	83.3	26.1	2.70	72.9	0.996	72.8

*Run not valid due to leak from broken glass filter.
(Break occurred after end of run)

Table 3-2. TEST DATA SUMMARY (CONTINUED)

Pacific Environmental Services
Page #2
ASARCO July, 1979

CTL - ENVIRONMENTAL SERVICES

LOCATION	RUN #	AND DATE	WEIGHT WATER COLLECTED grams	% WATER	PITOT TUBE CORRECTION FACTOR Cp	AVG. $\sqrt{\Delta P}$ Inches H_2O	AVG. STACK TEMPERATURE °F	STACK PRESSURE Inches Hg	DRY MOLECULAR WEIGHT STACK GAS	STACK VELOCITY Ft./Sec.
Main Stack	1	9-21-79	23.5	1.7	0.819	0.895	171	25.5	29.1	58.0
	2	9-21-79	31.4	3.1	0.819	0.891	176	25.5	29.1	58.1
	3	9-21-79	42.2	4.3	0.819	0.856	182	25.5	29.1	56.2
Flue to Zinc Stack	1	9-22-79	24.3	2.7	0.833	0.556	224	25.9	28.9	38.1
	2	9-23-79	12.0	1.3	0.833	0.579	211	25.9	28.9	39.1
	3	9-23-79	18.0	1.9	0.833	0.585	224	25.9	28.9	39.9
Bag House Stack #1	3	9-28-79	30.8	1.7	0.819	0.247	167	25.9	28.9	15.9
	4	9-28-79	26.2	1.6	0.819	0.221	178	25.9	28.9	14.3
	5	9-29-79	32.4	1.7	0.819	0.256	162	26.1	28.9	16.3
Bag House Stack #2	1	*9-26-79	29.6	1.8	0.839	0.219	160	25.9	28.9	14.3
	2	9-27-79	31.2	1.9	0.839	0.217	158	26.0	28.9	14.1
	3	9-28-79	25.5	1.5	0.839	0.217	149	25.9	28.9	14.0
	4	9-28-79	30.1	1.7	0.839	0.240	160	25.9	28.9	15.7
Bag House Stack #3	1	9-26-79	27.6	2.0	0.819	0.197	176	25.9	28.9	12.8
	2	9-27-79	28.2	1.9	0.819	0.197	168	26.0	28.9	12.6
	5	9-29-79	30.5	1.9	0.839	0.204	140	26.1	28.9	13.1

*Run not valid due to leak from broken glass filter.
(Break occurred after end of run)

Table 3-2. TEST DATA SUMMARY (CONTINUED)

Pacific Environmental Services
Page #3
ASARCO July, 1979

CTL - ENVIRONMENTAL SERVICES

LOCATION	RUN # AND DATE	NOZZLE DIAMETER Inches	SAMPLING TIME Minutes	% ISOKINETIC	FILTER WEIGHT GAIN mg	PROBE WASH AND IMPINGER CATCH mg	PROBE WASH AND IMPINGER CATCH VOLUME ml
Main Stack	1 9-21-79	0.250	84	93.1	28.7	179.	649
	2 9-21-79	0.250	60	95.1	27.4	161.	632
	3 9-21-79	0.250	60	95.3	21.1	153.	500
Flue to Zinc Stack	1 9-22-79	0.253	84	94.7	6.2	26.3	648
	2 9-23-79	0.253	84	95.1	4.7	34.4	718
	3 9-23-79	0.253	84	94.8	4.8	28.0	550
Bag House Stack #1	3 9-28-79	0.485	96	99.6	19.2	38.3	727
	4 9-28-79	0.485	96	100.	18.9	44.6	934
	5 9-28-79	0.485	96	99.8	15.2	32.3	604
Bag House Stack #2	1 *9-26-79	0.489	96	98.1	25.2	39.7	684
	2 9-27-79	0.489	96	99.1	21.4	20.7	586
	3 9-28-79	0.489	96	99.1	23.5	26.1	824
	4 9-28-79	0.485	96	97.4	29.6	56.8	559
Bag House Stack #3	1 9-26-79	0.485	96	98.2	18.4	53.4	535
	2 9-27-79	0.485	96	101.	15.1	21.3	711
	5 9-29-79	0.489	96	98.4	15.0	17.7	524

*Run not valid due to leak from broken glass filter.
(Break occurred after end of run)

Table 3-2. TEST DATA SUMMARY (CONTINUED)

Pacific Environmental Services
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ASARCO July, 1979

CTL - ENVIRONMENTAL SERVICES

LOCATION	RUN #	AND DATE	TOTAL WEIGHT LEAD COLLECTED mg				TOTAL WEIGHT CADMIUM COLLECTED mg				TOTAL WEIGHT ARSENIC COLLECTED mg			
			1	2	3	AVG	1	2	3	AVG	1	2	3	AVG
Main Stack	1	9-21-79	11.0	10.8	10.5	10.8	0.239	0.241	0.244	0.241	9.11	9.58	8.82	9.17
	2	9-21-79	11.8	11.2	11.5	11.5	0.183	0.180	0.185	0.183	11.6	11.6	10.9	11.4
	3	9-21-79	7.58	7.30	7.15	7.34	0.153	0.151	0.155	0.153	11.3	10.7	11.5	11.2
Flue to Zinc Stack	1	9-22-79	3.18	3.08	3.10	3.12	0.051	0.050	0.052	0.051	0.75	0.75	0.70	0.73
	2	9-23-79	1.06	1.05	1.03	1.05	0.019	0.020	0.017	0.019	0.47	0.41	0.45	0.44
	3	9-23-79	0.96	0.93	0.97	0.95	0.063	0.066	0.065	0.065	1.55	1.47	1.58	1.53
Bag House Stack #1	3	9-28-79	6.15	5.80	6.02	5.99	1.82	1.85	1.88	1.85	0.66	0.59	0.73	0.66
	4	9-28-79	6.10	6.02	6.07	6.06	1.63	1.61	1.62	1.62	1.48	1.38	1.40	1.42
	5	9-29-79	4.80	4.70	4.70	4.73	2.33	2.30	2.35	2.33	1.12	1.10	1.17	1.13
Bag House Stack #2	1	*9-26-79	8.73	8.40	8.48	8.54	2.29	2.30	2.28	2.29	0.57	0.54	0.54	0.55
	2	9-27-79	7.33	7.10	7.30	7.24	1.14	1.20	1.11	1.15	1.23	1.06	1.18	1.16
	3	9-28-79	5.38	5.40	5.41	5.40	1.54	1.55	1.53	1.54	0.72	0.77	0.68	0.72
	4	9-28-79	7.03	6.80	7.00	6.94	2.09	2.11	2.07	2.09	1.80	1.68	1.76	1.75
Bag House Stack #3	1	9-26-79	7.10	6.83	7.11	7.01	1.61	1.64	1.66	1.64	1.12	0.98	1.00	1.03
	2	9-27-79	5.00	5.01	4.90	4.97	0.74	0.76	0.75	0.75	1.04	1.04	0.95	1.01
	5	9-29-79	4.08	4.72	4.44	4.41	2.08	2.10	2.11	2.10	0.62	0.69	0.67	0.66

*Run not valid due to leak from broken glass filter.
(Break occurred after end of run)

Table 3-2. TEST DATA SUMMARY (CONCLUDED)

LOCATION	RUN	AND DATE	GAS VOLUME AT STACK CONDITIONS Dry Cubic Feet	STACK AREA Square Feet	LEAD MASS EMISSION RATE grams/day	CADMIUM MASS EMISSION RATE grams/day	ARSENIC MASS EMISSION RATE grams/day
Main Stack	1	9-21-79	91.5	56.2	33200	741	28200
	2	9-21-79	65.6	56.2	49600	789	49100
	3	9-21-79	62.9	56.2	32000	667	48800
Flue to Zinc Stack	1	9-22-79	61.6	99.4	16500	271	3870
	2	9-23-79	64.7	99.4	5450	98	2290
	3	9-23-79	65.4	99.4	4980	341	8030
Bag House Stack #1	3	9-28-79	115	127	9060	2800	998
	4	9-28-79	104	127	9130	2440	2140
	5	9-29-79	118	127	7140	3520	1710
Bag House Stack #2	1	*9-26-79	104	127	12900	3460	831
	2	9-27-79	103	127	10900	1720	1740
	3	9-28-79	103	127	8050	2230	1070
	4	9-28-79	113	127	10600	3180	2660
Bag House Stack #3	1	9-26-79	90.6	127	10800	2520	1590
	2	9-27-79	92.1	127	7460	1130	1520
	5	9-29-79	94.9	127	6650	3170	996

*Run not valid due to leak from broken glass filter.
(Break occurred after end of run)

APPENDIX

APPENDIX A

SAMPLING AND ANALYTICAL PROCEDURES

certified testing laboratories, inc.

SAMPLING AND ANALYTICAL PROCEDURES

All testing was performed with sampling equipment designed for isokinetic sampling to facilitate testing by EPA Standard Methods.

Gas flow rates were calculated using the observed gas temperature, molecular weight, pressure and velocity head measurements made with an S-type Pitot tube and a water manometer using Standard Method 2.

Moisture content was determined by passing a measured amount of gas through chilled impingers containing a known volume of deionized water, measuring the increase in weight of the impingers and of the silica gel used in the final drying of the gas, and calculating the amount of water vapor in the sample from this increase and the measured amount of gas.

The stack gas concentrations of CO_2, oxygen, CO, and nitrogen (by difference) were measured with a standard Orsat apparatus. These concentrations and the moisture content were used to determine molecular weight of the stack gas.

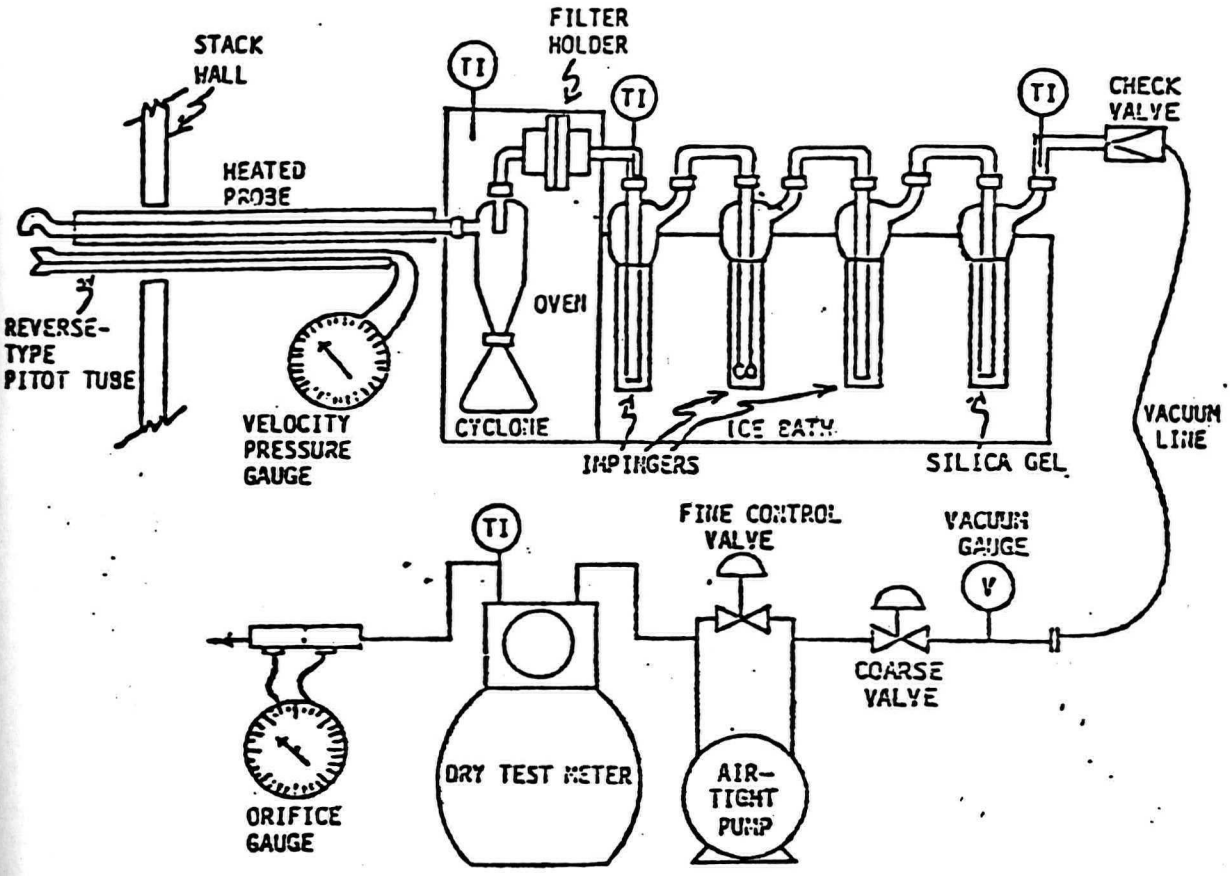
Standard Method 5 was used for determining particulate emissions. Measured stack gas samples were taken under isokinetic conditions. The samples were passed through a fiberglass filter, impingers, pump, a meter and an orifice as shown in the accompanying drawing, "Particulate Sampling Train".

The amount of filter catch is determined by the difference in the weight of the filter before and after the test, after desiccating. The particulate matter from other portions of the train was determined by rinsing the probe and all glassware ahead of the filter with 0.1N HNO_3, and evaporating to dryness along with the impinger catch and weighing.

The samples were analyzed for the requested elements after digestion, according to the EPA method for simultaneous determination of particulate and lead emissions. Lead and cadmium were analyzed by standard atomic absorption flame techniques, while arsenic was determined using the gaseous hydride technique. All samples were taken to a final volume of 250 ml and suitable aliquots taken for analysis.

APPENDIX B

DIAGRAM OF PARTICULATE SAMPLING TRAIN



PARTICULATE SAMPLING TRAIN

APPENDIX C

DESCRIPTION OF PROPOSED VARIATIONS TO EPA METHOD 5
(Letter: Salot to Gordon, with attachments)

certified testing laboratories, inc.

RECEIVED AUG 13 197

August 9, 1979

Mr. Bob Gordon
Pacific Environmental Services
1930 14th Street
Santa Monica, California 90404

Dear Bob:

Enclosed is the information you required for the stack test at ASARCO. Testing will be done in strict adherance to EPA Method 5, with the following possible exceptions:

1) Our Method 5 equipment is manufactured by Nutech Corporation, Durham, N. Carolina, and utilizes a silicon O-ring sealing system. (See enclosed drawing, figure 11.) The system has the advantage of not requiring silicon grease and produces a very good gas tight seal. Otherwise the impinger assembly is completely standard. (See figure 10.) It is my understanding from Nutech that the silicon O-ring configuration is currently being used by EPA itself.

2) Due to awkward placement of some parts, it may be necessary to utilize flexible teflon tubing between the probe assembly and the filter box. The teflon tubing is heavy-walled virgin teflon (not heat traced) and can be washed with acetone and brushed to retrieve probe washings. A 2-foot stainless steel tube is used to interface the teflon tubing to the heated filter box.

If you need any more information about either of these modifications, do not hesitate to contact me.

Analysis for lead, arsenic and cadmium will be carried out utilizing atomic absorption techniques. Lead and cadmium will be run either by flame or graphite furnace, depending on the concentration levels. Arsenic will be run by hydride generation. Detailed methods are included herein.

Our AA is a Perkin-Elmer, Model 460, and the graphite furnace is a Perkin Elmer HGA 2100.

Filter digestion will be carried out utilizing dilute nitric acid as specified in the Federal Register for Hi-Vol Lead samples, Volume 43, #194, October 1978.

Sincerely,

CERTIFIED TESTING LABORATORIES, INC.

Stuart E. Salot, Ph.D.

SES/bjh

2905 EAST CENTURY BLVD. • SOUTH GATE, CALIF. 90280 • (213) 564-2641

Glassware screw joints (Figure 11) require no silicone sealing grease. Frim, but not excessive, pressure should be used when forming a seal. Generally the screw joints need only be loosened and the male glass stems pushed in place. Occasionally it will be necessary to completely remove the screw cap from the threaded glass and push it over the male glass stem; refit and seal.

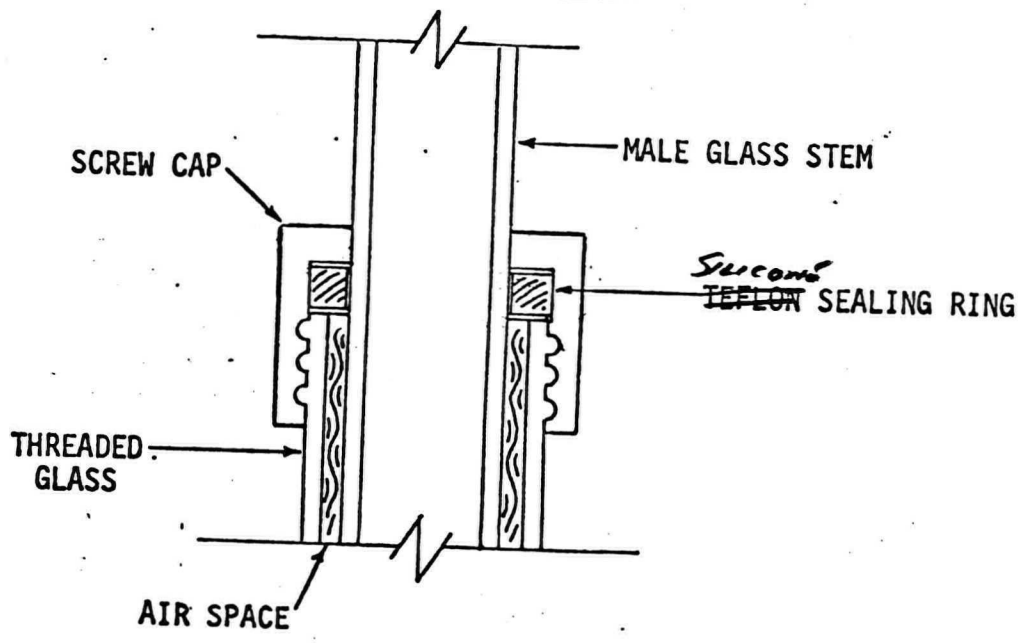


Figure 11. Glass screw joint.

2.2.4 Filter Assembly

Assemble the filter holder and components for a pressure-drop eck as shown in Figure 12. Turn the pump on and adjust flow the orifice gauge to a reading of 5.0 inches H_2O. If the cuum gauge on the control module reads higher than two inches mercury, the frit is dirty and should be cleaned and checked in.

IMPINGER GLASSWARE FOR
EPA PARTICULATE SAMPLING TRAIN
/WITH CYCLONE

Model NUE-S-100

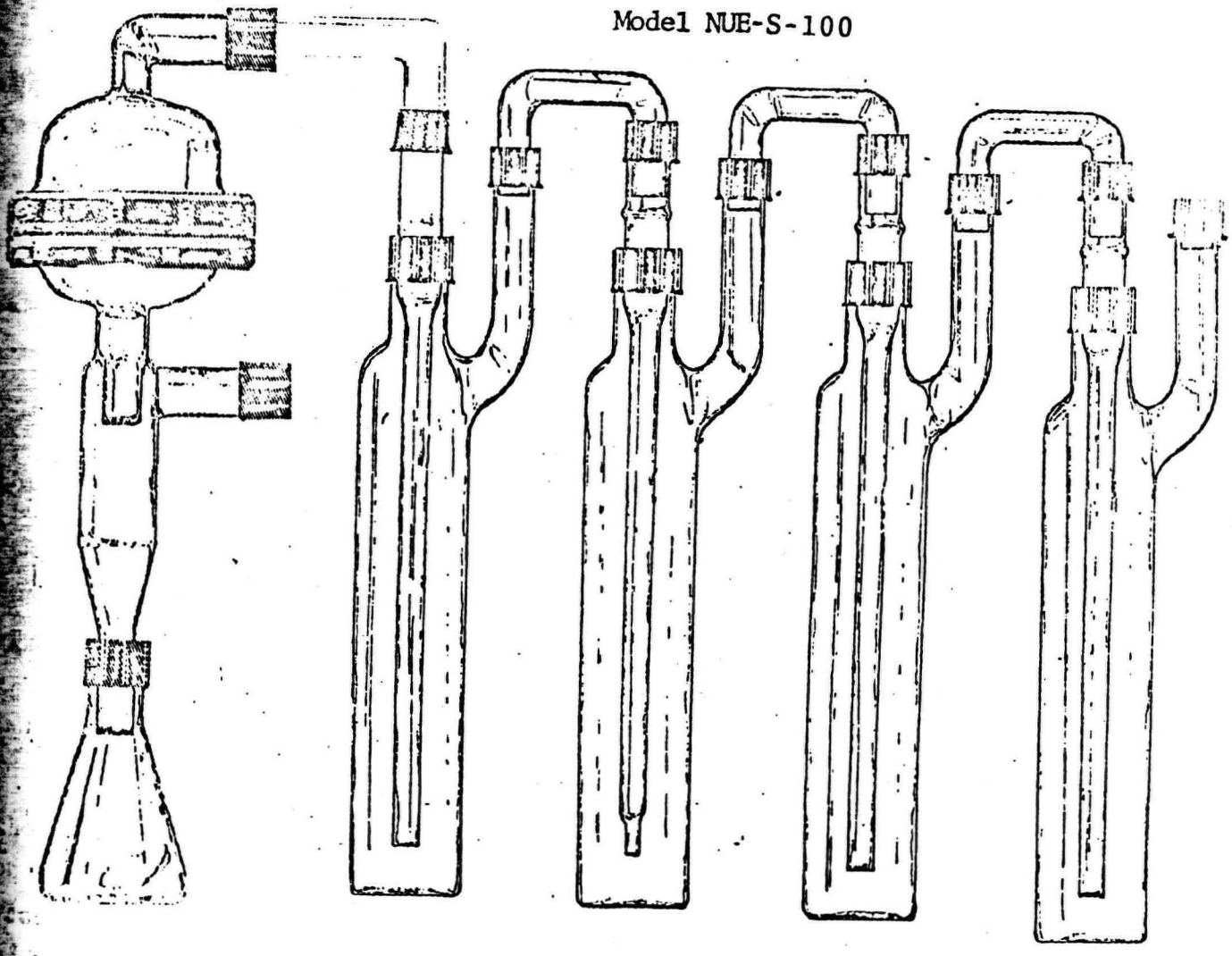


Figure 10. Impinger Glassware

HGA OPERATING PARAMETERS

Element	Anion Used	Wavelength nm	Slit as SBW nm	Maximum Charring Temp. (°C)	Optimum Atomization Temp. (°C)	Sensitivity (pg/0.0044 Abs) Purge Gas Flow		Comments
						Normal	Interrupted	
Li Lithium	Chloride	670.8 (335-VIS)	2.0/1.4	1000°	2700°	60.		
Mg Magnesium	Chloride	285.2	2.0	1200°	2000°	3.		
Mn Manganese	Chloride	279.5	0.2	1100°	2400°	8.	3.	
Mo Molybdenum	Molybdate	313.3	0.7	1800°	2700°	90.		
Na Sodium	Chloride	589.0 (295-VIS)	0.2/0.4	1200°	2000°	10.		
Nb Niobium		334.4	0.2					
Ni Nickel	Nitrate	232.0	0.2	1200°	2500°	140.		
Pb Lead	Nitrate	283.3	0.7	550°	2000°	50.	10.	See Note 1

HGA OPERATING PARAMETERS

Element	Anion Used	Wavelength nm	Slit as SBW nm	Maximum Charring Temp. (°C)	Optimum Atomization Temp. (°C)	Sensitivity (pg/0.0044 Abs) Purge Gas Flow		Comments
						Normal	Interrupted	
Cd Cadmium	Chloride	228.8	0.7	400°	1500°	2.	1.	
Co Cobalt	Chloride	240.7	0.2	1000°	2600°	80.	25.	
Cr Chromium	($KCrO_4$)	357.9	0.7	1350°	2700°	25.	10.	Possible CN absorption w/N_2 purge; use argon
Cs Cesium	Chloride	852.1 (426-VIS)	4.0	1200°	2400°	100.		Filter used
Cu Copper	Sulfate	324.7	0.7	1000°	2500°	50.		
Dy Dysprosium	Nitrate	421.2	0.2	1400°	2700°	660.		
Er Erbium	Chloride	400.8	0.2	1400°	2700°	1,660.		
Eu Europium	Chloride	459.4 (230-VIS)	0.2/0.4	1400°	2700°	40,000.		

STANDARD CONDITIONS FOR CADMIUM

STOCK STANDARD SOLUTION

Cadmium, 1000 µg/ml. Dissolve 1.000 g of cadmium metal in a minimum volume of (1+1) HCl. Dilute to 1 liter with 1% (v/v) HCl.

OPERATING PARAMETERS

Perkin-Elmer Instrument Settings

Instrument	X03 series	X60, X00 series	X90 series
Wavelength 228.8 nm	229 - UV	229	110
Slit Setting	4 (0.7 nm)	0.7 nm	0.7 nm
Light Source	Electrodeless Discharge Lamp or Hollow Cathode Lamp		
Flame Type	Air-acetylene flame Oxidizing (lean, blue)		

SENSITIVITY

For the standard conditions described above, the sensitivity is about 0.025 µg/ml Cd for 1% absorption. A standard containing 2 µg/ml Cd will typically give an absorbance reading of about 0.35 absorbance units (about 55% absorption).

LINEAR WORKING RANGE

For the standard conditions described above, the working range for Cd is linear up to concentrations of approximately 2 µg/ml in aqueous solution.

OTHER ANALYTICAL LINES

Wave-length	X03 series	X60, X00 series	X90 series	Slit	Relative Sensitivity
228.8 nm	229 - UV	229	110	0.7 nm	1.
326.1 nm	326 - UV	326	283	0.7 nm	435.

OTHER FLAMES

The nitrous oxide-acetylene flame will provide a sensitivity of about 0.1 µg/ml for 1% absorption.

LIGHT SOURCES

Both Electrodeless Discharge Lamps (EDL's) and Hollow Cathode Lamps are available for cadmium. EDL's provide greater light output and longer life than Hollow Cathode Lamps. For cadmium, both EDL's and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

MICROSAMPLING SYSTEM

With the Sampling Boat, about 0.0001 µg of cadmium can be detected. With the Delves Sampling Cup Technique a detection limit of 0.00005 µg of cadmium may be achieved. Refer to the General Information section for additional details on the use of the AA Microsampling System.

FLAME EMISSION

The most sensitive emission wavelength for cadmium is at 326.1 nm. A nitrous oxide-acetylene flame is recommended. Cadmium can also be determined at the 228.8 nm wavelength, but with reduced sensitivity. An air-acetylene flame may also be used with reduced sensitivity.

STANDARD CONDITIONS FOR LEAD

STOCK STANDARD SOLUTION

Lead, 1000 µg/ml. Dissolve 1.598 g of lead nitrate, $Pb(NO_3)_2$, in 1% (v/v) HNO_3 and dilute to 1 liter with 1% (v/v) HNO_3.

OPERATING PARAMETERS

Perkin-Elmer Instrument Settings

Instrument	X03 series	X60, X00 series	X90 series
Wavelength 283.3 nm	283 - UV	283	206
Slit Setting	4 (0.7 nm)	0.7 nm	0.7 nm
Light Source	Electrodeless Discharge Lamp or Hollow Cathode Lamp		
Flame Type	Air-acetylene flame Oxidizing (lean, blue)		

20ppm = .20 6/15/78

SENSITIVITY

For the standard conditions described above, the sensitivity is about 0.5 µg/ml Pb for 1% absorption. A standard containing 20 µg/ml Pb will typically give an absorbance reading of about 0.18 absorbance units (about 34% absorption).

LINEAR WORKING RANGE

For the standard conditions described above, the working range for Pb is linear up to concentrations of approximately 20 µg/ml in aqueous solution.

OTHER ANALYTICAL LINES

Wavelength	X03 series	X60, X00 series	X90 series	Slit	Relative Sensitivity
283.3 nm	283 - UV	283	206	0.7 nm	1.0
217.0 nm	217 - UV	217	089	0.7 nm	0.4
261.4 nm	261 - UV	261	167	0.7 nm	10.
368.4 nm	368 - UV	368	359	0.7 nm	25.

OTHER FLAMES

The nitrous oxide-acetylene flame will provide a sensitivity of about 1.7 µg/ml for 1% absorption.

LIGHT SOURCES

Both Electrodeless Discharge Lamps (EDL's) and Hollow Cathode Lamps are available for lead. EDL's provide greater light output and longer life than Hollow Cathode Lamps. For lead, both EDL's and Hollow Cathode Lamps provide approximately the same sensitivity and detection limit.

With multi-element lamps containing copper, the Cu 216.5 nm resonance line may interfere with lead determinations at the lead 217.0 nm line. The lead 283.3 nm line should be used instead.

MICROSAMPLING SYSTEM

With the Sampling Boat, about 0.001 µg of lead can be detected. Refer to the General Information section for additional details on the use of the Sampling Boat. With the Delves Sampling Cup technique, about 0.0001 µg of lead can be detected.

FLAME EMISSION

The most sensitive emission wavelength for lead is at 405.8 nm. A nitrous oxide-acetylene flame is recommended. Lead can also be determined at the 368.4, 283.3, and 261.4 nm wavelengths, but with reduced sensitivity. An air-acetylene flame may also be used with reduced sensitivity.

Reprinted from Atomic Absorption Newsletter Vol. 12, No. 4

ATOMIC ABSORPTION DETERMINATION OF GASEOUS HYDRIDES UTILIZING SODIUM BOROHYDRIDE REDUCTION

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ABSTRACT

A method for the determination of As, Bi, Ge, Sb, Se, Sn and Te by reduction to the corresponding hydride is described. The gaseous hydride, generated by reduction with sodium borohydride, is collected in a balloon reservoir attached to the generation flask, and subsequently swept by a flow of argon into an argon-hydrogen-entrained air flame. Optimum parameters, including acid concentration and collection time, were determined for each element. Sensitivity, linearity, precision, and detection limits were measured. The method is simple, relatively rapid, and provides absolute detection limits in the ng range.

RESUME

Une méthode pour la détermination d'As, Bi, Ge, Sb, Se et Sn par réduction de leurs hydrures correspondants est décrite. Les hydrures gazeux, générés par réduction avec du borohydrate de sodium sont collectés dans un ballon reservoir connecté au flaçon de génération et ensuite entrainés par de l'argon dans une flamme air/argon/hydrogene. Les paramètres optimums incluant la concentration en acide et le temps de génération sont déterminés par chaque élément. La méthode est simple, relativement rapide et fournit une détection limite absolue au niveau du monogramme.

ZUSAMMENFASSUNG

Eine Methode zur Bestimmung von As, Bi, Ge, Sb, Se, Sn und Te durch Reduktion zum entsprechenden Hydrid wird beschrieben. Das gasförmige Hydrid, das durch Reduktion mit Natrium-borhydrid erzeugt wird, wird in einem an den Reaktionskolben angeschlossenen Vorratsballon gesammelt und schliesslich durch einen Argonstrom in eine Argon/Wasserstoff-Diffusionsflamme gespült. Für jedes Element wurden die optimalen Parameter einschliesslich Säurekonzentration und Reaktionszeit bestimmt. Die Empfindlichkeit, Linearität, Präzision und Nachweisgrenze wurden gemessen. Das Verfahren ist einfach, relativ schnell und liefert absolute Nachweisgrenzen im ng-Bereich.

INTRODUCTION

The determination of As and Se by hydride generation and subsequent analysis by atomic absorption has been described by several authors (1-11). Results have been reported using Zn metal and $SnCl_2$ (1-8) and Mg metal and $TiCl_3$ (9, 10) as reductants and sources of nascent hydrogen. Pollock and West (9, 10) have also determined Bi, Sb, and Te utilizing Mg-$TiCl_3$ reduction. Schmidt and Royer (11) have recently reported the determination of As, Bi, Sb and Se utilizing reduction with $NaBH_4$. The determination of Ge using $NaBH_4$ reduction has been reported by Pollock and West (10).

The present paper describes the use of $NaBH_4$ as the reductant for the determination of As, Bi, Ge, Sb, Se, Sn and Te by hydride generation. The gaseous hydride is collected in a balloon reservoir and subsequently swept by an argon carrier gas into an argon-hydrogen-entrained air flame.* Experiments were performed to determine the optimum acid concentration and hydride collection time. Standard curves were prepared, and linearity, sensitivity, precision and detection limits were measured for each of the elements studied.

*A nitrogen-hydrogen-entrained air flame, with nitrogen carrier gas, gave equal performance to argon-hydrogen for the determination of As as arsine.

EXPERIMENTAL

All data were obtained using a Perkin-Elmer Model 403 atomic absorption spectrophotometer equipped with a 3-slot burner head, Deuterium Background Corrector (12*f*, Model 056 recorder, and Intensitron hollow cathode lamps. For the determination of As and Se, Perkin-Elmer Electrodeless Discharge Lamps (13) were also employed. A Perkin-Elmer As/Se Sampling System® (5) was utilized for the generation and collection of the gaseous hydrides. This system utilizes a dosing stopcock for reagent introduction and a balloon reservoir for collection of the generated gases. By rotating a 4-way stopcock, the argon flow can be set to bypass or flow through the generation flask (125 ml Erlenmeyer with a 29/42 ground glass joint). The collected hydride, plus excess hydrogen, is introduced into the burner via the auxiliary oxidant connection. The following flow settings were found to be optimum: argon 40 (13 liters/min.) at a pressure of 20 psi, hydrogen 24 (10 liters/min.) at a pressure of 20 psi. Reagents used

were: $NaBH_4$ pellets (10 32", available from Alfa Inorganics*), and hydrochloric acid. Standard solutions of all the elements investigated were prepared in dilute HCl.

PROCEDURE

The procedure used for the generation of the gaseous hydrides is as follows:

1. Pipet 20 ml of sample into the generation flask.
2. Acidify the sample using a suitable volume of HCl (see discussion) and dilute to 40 ml with deionized water.
3. Connect the flask to the generation apparatus and open the 4-way stopcock for about 15 seconds to admit argon, which flushes the air out of the system.
4. After flushing, close the 4-way stopcock and add a single $NaBH_4$ pellet (10/32") via the dosing stopcock.
5. The reaction is allowed to continue for a time that will vary depending on the element being determined (see discussion), and the type of sample being analyzed. For aqueous samples, a reaction time of 30 seconds is suitable. Longer reaction times may be required for some types of samples.
6. Open the 4-way stopcock, which allows the auxiliary argon flow to sweep the generated gases into the burner. Record the absorption signal on a recorder.
7. Close the 4-way stopcock after the absorption signal has been recorded, and the pen has returned to the baseline.
8. Standards, including a reagent blank, are analyzed using the same procedure.

When the 4-way stopcock is opened, the surge of excess hydrogen into the flame causes a sudden change in the absorption of the flame, which produces a large blank signal when operating at wavelengths below 210 nm. Use of the D_2 Background Corrector (12) appreciably reduces this blank signal. All of the data reported in this study were obtained using the D_2 Background Corrector.

DISCUSSION

Initial studies were performed to establish what elements could be determined via hydride generation with $NaBH_4$ reduction. Using the procedure outlined, the following elements were investigated: As, Bi, Ga, Ge, Pb, Se, Sn and Te by atomic absorption plus P and S by molecular flame emission. Atomic absorption signals were obtained for the hydrides of all of the elements listed, with the exception of Ga and Pb. No absorption signals were obtained for Ga (287 nm) and Pb (283 nm) at levels up to 1000 µg. Attempts to determine P and S by molecular flame emission were also unsuccessful. The sudden change in flame emission caused by the introduction of the generated gases, produced off-scale blank readings at the HPO (526 nm) and S_2 (394 nm) wavelengths.

REACTION AND COLLECTION TIMES

The effect of hydride collection time (time period between adding the $NaBH_4$ and sweeping the generated gases into the flame) was investigated for As, Bi, Ge, Sb, Se, Sn and Te. Only for the analysis of Bi and Te is there a need for careful monitoring of the collection time. Figure 1 shows tracings obtained for 2 µg of Te (H_2Te) using various collection times, ranging from 15 seconds to 3 minutes. A significant loss of sensitivity is obtained using collection times longer than 30 seconds. This agrees with Pollock and West (10), who have reported that the Te sensitivity is affected by the collection time when using Mg-$TiCl_3$ reduction. By keeping the 4-way stopcock in the sweep position, one may continuously flush the H_2Te into the burner. This degrades the sensitivity by about a factor of 2; however, the need for time monitoring is eliminated.

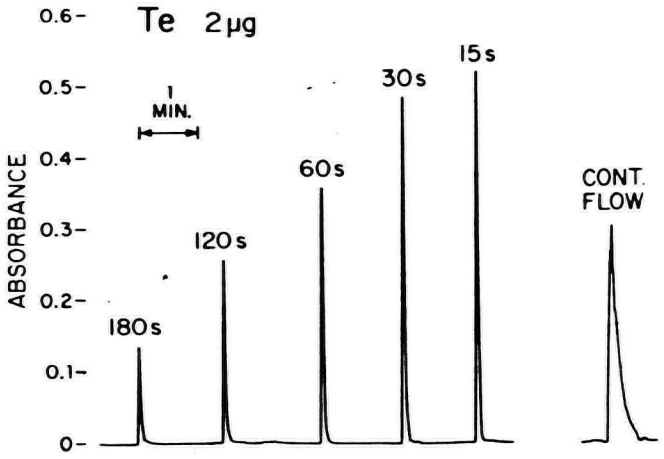

Fig. 1. Signals obtained for 2 µg Te, showing the effect of hydride collection time on sensitivity.

Calibration curves for Te, obtained using both continuous flow and a 30-second collection period, are shown in Figure 2. Similar calibration curves for Bi (H_3Bi) are shown in Figure 3. For Bi, optimum sensitivity is obtained using a 30-second collection period. The use of longer collection periods resulted in up to a 2-fold loss of sensitivity. The sensitivity obtained allowing the H_3Bi to flow continuously into the burner is about 50% poorer than that obtained by collecting the generated gases for 30 seconds.

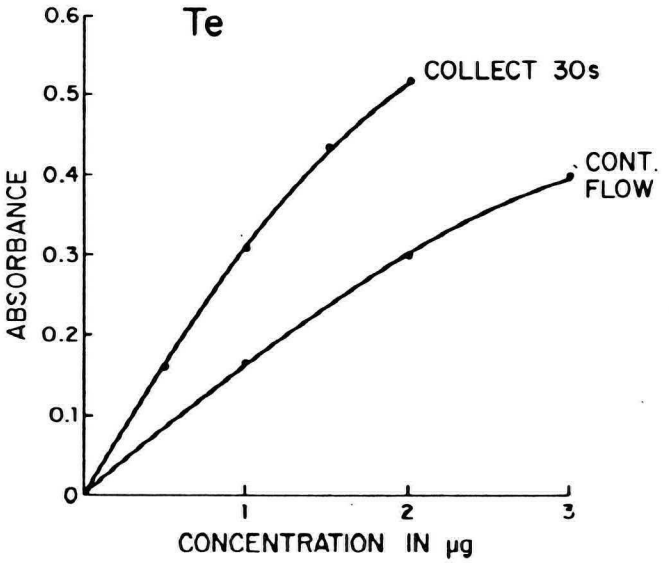

Fig. 2. Calibration curves for Te, obtained using a 30 second collection period, and continuous flow.

*Division of Ventron Corporation, Beverly, Mass. 01915.

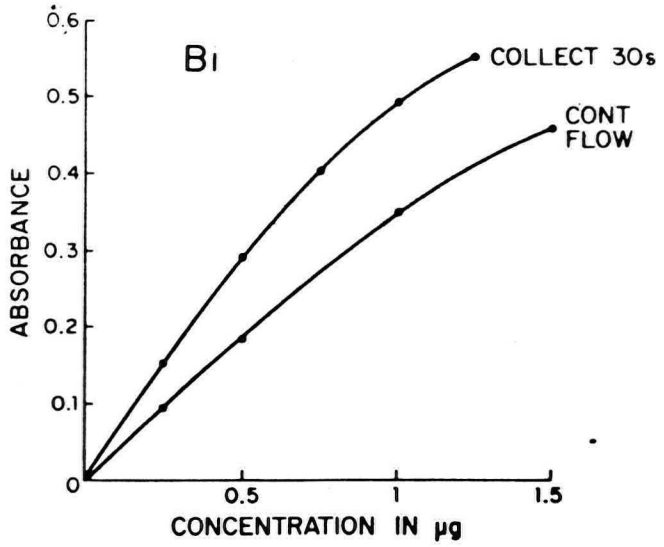

Fig. 3. Calibration curves for Bi, obtained using a 30 second collection period, and continuous flow.

For the other elements determined successfully using the $NaBH_4$ method (As, Ge, Sb, Se and Sn), collection times of up to 2 - 3 minutes can be used with no loss of sensitivity. Generally, a collection time of 30 seconds is sufficient, although longer collection times may be required for certain types of samples. The $NaBH_4$ pellet reacts vigorously when added to the acidified sample, and dissolves completely in about 20 seconds. The addition of a single $NaBH_4$ pellet (10/32"), weighing approximately 200 mg was found to provide optimum results. Varying the amount of $NaBH_4$ from 100 to 400 mg had no noticeable effect on the sensitivity obtained for any of the elements studied. The addition of amounts larger than 500 mg caused the balloon reservoir to rupture. The use of a magnetic stirrer to agitate a sample solution of As had no effect on the sensitivity obtained.

ACIDITY

The relationship of hydrochloric acid concentration to sensitivity is shown in Figure 4. For As, Bi, Sb and Te, varying the acid concentration from 1 to 6N had no noticeable effect on the sensitivity obtained. To obtain optimum sensitivity, the acid concentration should be at least 5N when determining Se, and 1N or less when determining Ge. For the determination of Sn, it is important that the acid concentration be 0.5N or less, due to the very pronounced loss of sensitivity with increasing acid concentration. The suitability of other acids was not investigated.

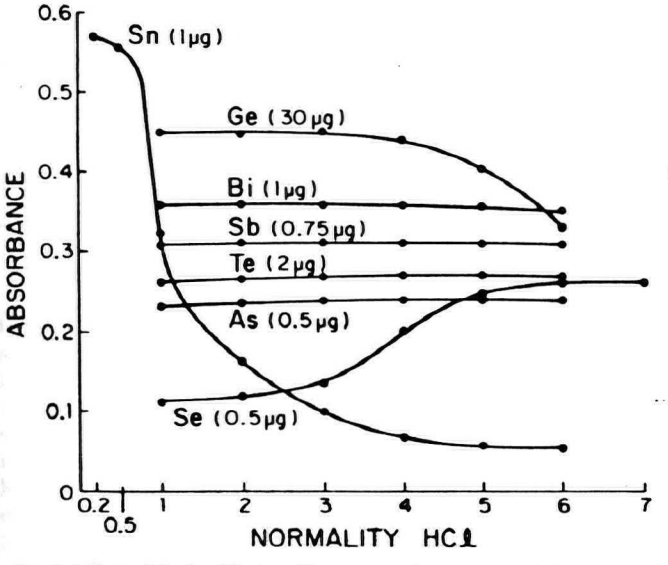

Fig. 4. Effect of hydrochloric acid concentration on sensitivity.

SENSITIVITY, PRECISION AND DETECTION LIMITS

Calibration curves for As (AsH_3) and Se (H_2Se), obtained using both hollow cathode lamps and electrodeless discharge lamps (EDL's) are shown in Figure 5. For As, the EDL provides about a 2-fold improvement in sensitivity while for Se the sensitivity is improved by about 30%. Figure 6 shows calibration curves obtained for Sn (SnH_4), Sb (H_3Sb) and Ge (GeH_4). The sensitivity for Ge is about an order of magnitude poorer that that obtained for the other elements studied. The absolute sensitivities (weight of an element which gives a signal of 1% absorption) obtained for the 7 elements studied are summarized in Table I.

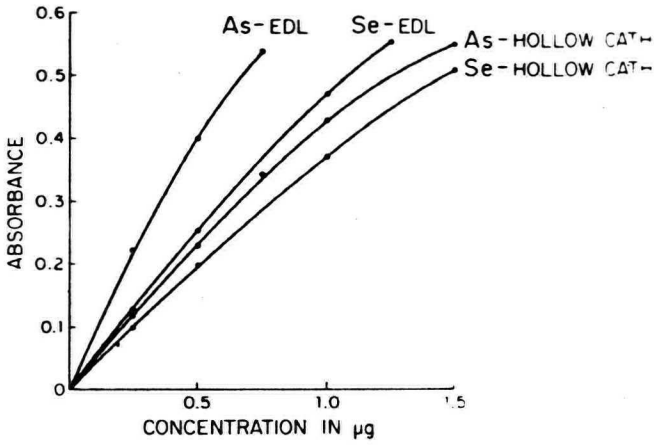

Fig. 5. Calibration curves for As and Se, obtained using hollow cathode lamps and EDL's.

TABLE I
Absolute Sensitivities Obtained Utilizing $NaBH_4$ Reduction

Element	*λ (nm)*	*Spectral Slit (nm)*	*Absolute Sensitivity (ng)*	*Remarks*
As	194	0.7	10	hollow cathode
			5	EDL
Bi	223	0.2	8	collect 30 s
			12	continuous flow
Ge	265	0.2	270	
Sb	218	0.2	10	
Se	196	0.7	11	hollow cathode
			9	EDL
Sn	224	0.2	7	
Te	214	0.2	14	collect 30 s
			27	continuous flow

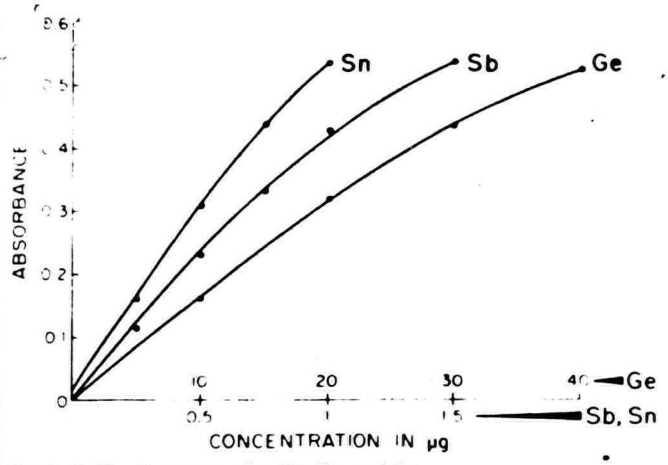

Fig. 6. Calibration curves for Ge, Sb, and Sn.

The repeatability of the $NaBH_4$ method was examined for each of the elements investigated. Ten replicate determinations of a standard providing an absorbance of 0.15 to 0.35 were made, and the resultant coefficient of variation calculated. The data obtained are summarized in Table II. For Bi and Te, precision was measured by collecting the generated gases for 30 seconds, and also by continuously flushing the gases into the burner. Both procedures provided equivalent precision. The precision obtained for Ge and Sn is poorer than that obtained for the other elements studied. Pollock and West (10) reported relatively poor precision determining Ge by $NaBH_4$-H_2SO_4 reduction.

TABLE II
Precision Summary

Element	*Weight in µg*	*C.V.* (%)*	*Remarks*
As	0.5	3.4	hollow cathode
	0.25	3.2	EDL
Bi	0.5	2.7	collect 30 s
	0.5	3.0	continuous flow
Ge	15	6.1	
Sb	0.5	3.6	
Se	0.5	2.9	hollow cathode
	0.5	3.1	EDL
Sn	0.5	6.7	
Te	1	3.8	collect 30 s
	2	4.1	continuous flow

*Coefficient of variation based on 10 replicate determinations.

Comparison recorder tracings taken near the As detection limit with a hollow cathode lamp and EDL, are shown in Figure 7. The much greater intensity provided by the EDL (13) enables one to operate at a much lower instrument gain setting; consequently, more scale expansion can be utilized. For As, the absolute detection limit is about 3 ng with the EDL, versus about 10 ng with the hollow cathode lamp. Figure 8 shows tracings for Te at detection limit levels. The signals were obtained by collecting the generated gases for 30 seconds, and also by continuously flushing the gases into the burner. Collecting

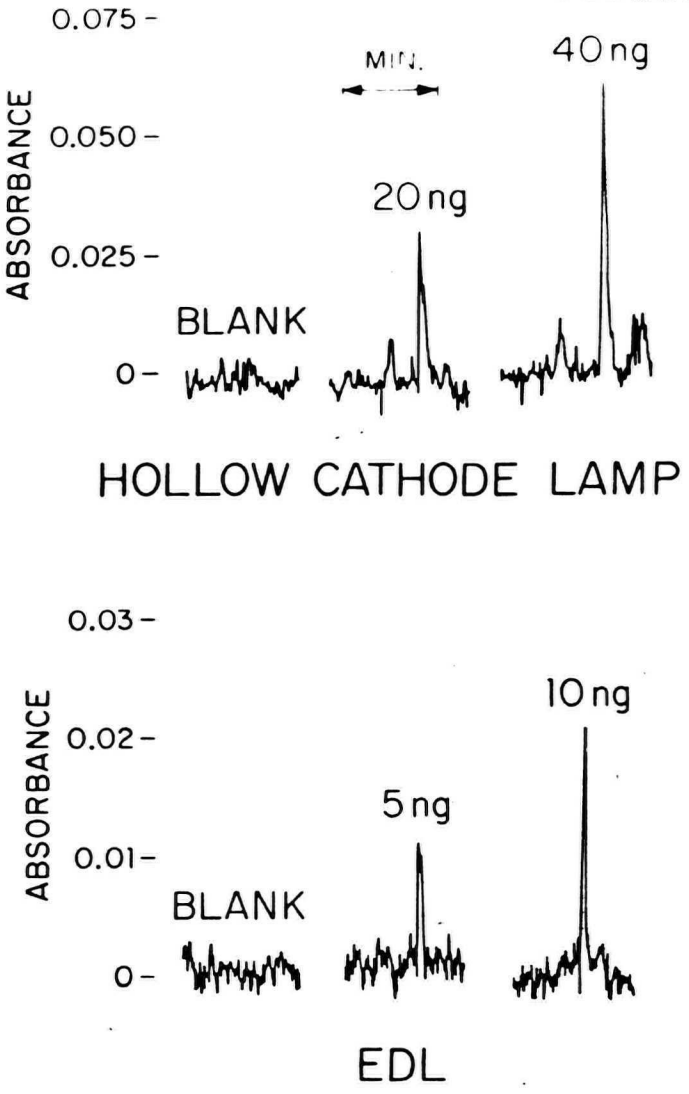

Fig. 7. Detection limit tracings for As, obtained using a hollow cathode lamp and EDL.

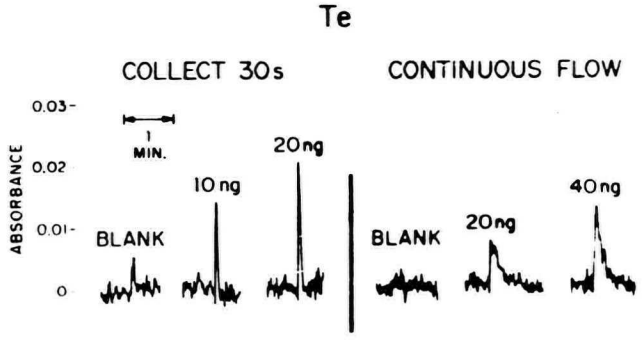

Fig. 8. Detection limit tracings for Te, obtained using a 30-second collection time, and continuous flow.

the hydride provides an absolute detection limit of about 5 ng, versus a value of about 15 ng obtained with continuous flow. Detection limits, measured for all of the elements studied, are summarized in Table III. The solution detection limits, measured using 20 ml of sample, can be improved still further by increasing the sample volume.

TABLE III
Detection Limit Summary

Element	Absolute Detection Limit (μg)	Solution Detection Limit* (μg/liter)	Remarks
As	0.010	0.5	hollow cathode
	0.003	0.15	EDL
Bi	0.005	0.25	collect 30 s
	0.008	0.4	continuous flow
Ge	0.2	10	
Sb	0.005	0.25	
Se	0.005	0.25	hollow cathode
	0.003	0.15	EDL
Sn	0.004	0.2	
Te	0.005	0.25	collect 30 s
	0.015	0.75	continuous flow

*Based on a 20-ml sample volume.

INTERFERENCES

Interferences were not investigated in this study; however, those materials that interfere with hydride formation in classical analytical methods (14 - 16) would be expected to cause interference with this method as well.

GENERAL

The $NaBH_4$ method offers several advantages over the Zn-$SnCl_2$ procedure for hydride generation. The $NaBH_4$ pellets are inexpensive (~2¢ per pellet), easy to handle and are sufficiently uniform to eliminate the need for weighing. By comparison, the Zn granules must be weighed, and a longer reaction time (2 - 3 minutes) is required. The zinc granules also tend to stick to the dosing stopcock, which is made of Teflon (5). If care is not exercised, the zinc granules can score the Teflon stopcock, causing the system to leak. The $NaBH_4$ pellets eliminate this problem. Another advantage of the $NaBH_4$ method is the very low blank signals which were obtained for the 7 elements studied. It is difficult to obtain As-free $SnCl_2$ which is used in the Zn reduction method.

CONCLUSIONS

The generation of gaseous hydrides by reduction with $NaBH_4$ is rapid and precise. Using the procedure outlined, As, Bi, Ge, Sb, Se, Sn and Te were successfully determined, with detection limits in the ng range. The $NaBH_4$ method offers several advantages over the Zn-$SnCl_2$ method for hydride generation.

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Received May 22, 1973

of HNO_3 and hydrochloric acid (HCl) facilitated by ultrasonication.

1.3 The lead content of the sample is analyzed by atomic absorption spectrometry using an air-acetylene flame, the 283.3 or 217.0 nm lead absorption line, and the optimum instrumental conditions recommended by the manufacturer.

1.4 The ultrasonication extraction with HNO_3/HCl will extract metals other than lead from ambient particulate matter.

2. *Range, sensitivity, and lower detectable limit.* The values given below are typical of the methods capabilities. Absolute values will vary for individual situations depending on the type of instrument used, the lead line, and operating conditions.

2.1 Range. The typical range of the method is 0.07 to 7.5 μg Pb/m^3 assuming an upper linear range of analysis of 15 μg/ml and an air volume of 2,400 m^3.

2.2 *Sensitivity.* Typical sensitivities for a 1 percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 μg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

2.3 Lower detectable limit (LDL). A typical LDL is 0.07 μg Pb/m^3. The above value was calculated by doubling the between-laboratory standard deviation obtained for the lowest measurable lead concentration in a collaborative test of the method.(*15*) An air volume of 2,400 m^3 was assumed.

3. *Interferences.* Two types of interferences are possible: chemical and light scattering.

3.1 *Chemical.* Reports on the absence (*1, 2, 3, 4, 5*) of chemical interferences far outweigh those reporting their presence, (*6*) therefore, no correction for chemical interferences is given here. If the analyst suspects that the sample matrix is causing a chemical interference, the interference can be verified and corrected for by carrying out the analysis with and without the method of standard additions.(*7*)

3.2 Light scattering. Nonatomic absorption or light scattering, produced by high concentrations of dissolved solids in the sample, can produce a significant interference, especially at low lead concentrations. (*2*) The interference is greater at the 217.0 nm line than at the 283.3 nm line. No interference was observed using the 283.3 nm line with a similar method.(*1*)

Light scattering interferences can, however, be corrected for instrumentally. Since the dissolved solids can vary depending on the origin of the sample, the correction may be necessary, especially when using the 217.0 nm line. Dual beam instruments with a continuum source give the most accurate correction. A less accurate correction can be obtained by using a nonabsorbing lead line that is near the lead analytical line. Information on use of these correction techniques can be obtained from instrument manufacturers' manuals.

If instrumental correction is not feasible, the interference can be eliminated by use of the ammonium pyrrolidinecarbodithioate-methylisobutyl ketone, chelation-solvent extraction technique of sample preparation.(*8*)

4. *Precision and bias.*

4.1 The high-volume sampling procedure used to collect ambient air particulate matter has a between-laboratory relative standard deviation of 5.7 percent over the range 80 to 125 $\mu g/m^3$.(*9*) The combined extraction-analysis procedure has an average within-laboratory relative standard deviation of 5 to 6 percent over the range 1.5 to 15 μg Pb/ml, and an average between laboratory relative standard deviation of 7 to 9 percent over the same range. These values include use of either extraction procedure.

4.2 Single laboratory experiments and collaborative testing indicate that there is no significant difference in lead recovery between the hot and ultrasonic extraction procedures.(*15*)

5. *Apparatus.*

5.1 Sampling.

5.1.1 High-volume sampler. Use and calibrate the sampler as described in reference 10.

5.2 Analysis.

5.2.1 Atomic absorption spectrophotometer. Equipped with lead hollow cathode or electrodeless discharge lamp.

5.2.1.1 Acetylene. The grade recommended by the instrument manufacturer should be used. Change cylinder when pressure drops below 50–100 psig.

5.2.1.2 Air. Filtered to remove particulate, oil, and water.

5.2.2 Glassware. Class A borosilicate glassware should be used throughout the analysis.

5.2.2.1 Beakers. 30 and 150 ml, graduated, Pyrex.

5.2.2.2 Volumetric flasks. 100-ml.

5.2.2.3 Pipettes. To deliver 50, 30, 15, 8, 4, 2, 1 ml.

5.2.2.4 Cleaning. All glassware should be scrupulously cleaned. The following procedure is suggested. Wash with laboratory detergent, rinse, soak for 4 hours in 20 percent (w/w) HNO_3, rinse 3 times with distilled-deionized water, and dry in a dust free manner.

5.2.3 Hot plate.

5.2.4. Ultrasonication water bath, unheated. Commercially available laboratory ultrasonic cleaning baths of 450 watts or higher "cleaning power," i.e., actual ultrasonic power output to the bath have been found satisfactory.

5.2.5 Template. To aid in sectioning the glass-fiber filter. See figure 1 for dimensions.

5.2.6 Pizza cutter. Thin wheel. Thickness <1mm.

5.2.7 Watch glass.

5.2.8 Polyethylene bottles. For storage of samples. Linear polyethylene gives better storage stability than other polyethylenes and is preferred.

5.2.9 Parafilm "M".* American Can Co., Marathon Products, Nennah, Wis., or equivalent.

6. *Reagents.*

6.1 Sampling.

6.1.1 Glass fiber filters. The specifications given below are intended to aid the user in obtaining high quality filters with reproducible properties. These specifications have been met by EPA contractors.

6.1.1.1 Lead content. The absolute lead content of filters is not critical, but low values are, of course, desirable. EPA typically obtains filters with a lead content of <75 μg/filter.

It is important that the variation in lead content from filter to filter, within a given batch, be small.

6.1.1.2 Testing.

6.1.1.2.1 For large batches of filters (>500 filters) select at random 20 to 30 filters from a given batch. For small batches (<500 filters) a lesser number of filters may be taken. Cut one 3/4"x8" strip from each filter anywhere in the filter. Analyze all strips, separately, according to the directions in sections 7 and 8.

6.1.1.2.2 Calculate the total lead in each filter as

$$F_b = \mu g\ Pb/ml \times \frac{100\ ml}{strip} \times \frac{12\ strips}{filter}$$

where:

F_b = Amount of lead per 72 square inches of filter, μg.

6.1.1.2.3 Calculate the mean, F_b, of the values and the relative standard deviation (standard deviation/mean × 100). If the relative standard deviation is high enough so that, in the analysts opinion, subtraction of F_b (section 10.3) may result in a significant error in the μg Pb/m^3 the batch should be rejected.

6.1.1.2.4 For acceptable batches, use the value of F_b to correct all lead analyses (section 10.3) of particulate matter collected using that batch of filters. If the analyses are below the LDL (section 2.3) no correction is necessary.

6.2 Analysis.

6.2.1 Concentrated (15.6 *M*) HNO_3. ACS reagent grade HNO_3 and commercially available redistilled HNO_3 has found to have sufficiently low lead concentrations.

6.2.2 Concentrated (11.7 *M*) HCl. ACS reagent grade.

6.2.3 Distilled-deionized water. (D.I. water).

6.2.4 3 *M* HNO_3. This solution is used in the hot extraction procedure. To prepare, add 192 ml of concentrated HNO_3 to D.I. water in a 1 *l* volumetric flask. Shake well, cool, and dilute to volume with D.I. water. *Caution:* Nitric acid fumes are toxic. Prepare in a well ventilated fume hood.

6.2.5 0.45 *M* HNO_3. This solution is used as the matrix for calibration standards when using the hot extraction procedure. To prepare, add 29 ml of concentrated HNO_3 to D.I. water in a 1 *l* volumetric flask. Shake well, cool, and dilute to volume with D.I. water.

6.2.6 2.6 *M* HNO_3 + 0 to 0.9 *M* HCl. This solution is used in the ultrasonic extraction procedure. The concentration of HCl can be varied from 0 to 0.9 *M*. Directions are given for preparation of a 2.6 *M* HNO_3 + 0.9 *M* HCl solution. Place 167 ml of concentrated HNO_3 into a 1 *l* volumetric flask and add 77 ml of concentrated HCl. Stir 4 to 6 hours, dilute to nearly 1 *l* with D.I. water, cool to room temperature, and dilute to 1 *l*.

6.2.7 0.40 *M* HNO_3 + X *M* HCl. This solution is used as the matrix for calibration standards when using the ultrasonic extraction procedure. To prepare, add 26 ml of concentrated HNO_3, plus the ml of HCl required, to a 1 *l* volumetric flask. Dilute to nearly 1 *l* with D.I. water, cool to room temperature, and dilute to 1 *L*. The amount of HCl required can be determined from the following equation:

$$y = \frac{77\ ml \times 0.15 \times}{0.9\ M}$$

*Mention of commercial products does not imply endorsement by the U.S. Environmental Protection Agency.

where:

y = ml of concentrated HCl required.
x = molarity of HCl in 6.2.6.
0.15 = dilution factor in 7.2.2.

6.2.8 Lead nitrate, $Pb(NO_3)_2$. ACS reagent grade, purity 99.0 percent. Heat for 4 hours at 120° C and cool in a desiccator.

6.3 Calibration standards.

6.3.1 Master standard, 1000 μg Pb/ml in HNO_3. Dissolve 1.598 g of $Pb(NO_3)_2$ in 0.45 *M* HNO_3 contained in a 1 *l* volumetric flask and dilute to volume with 0.45 *M* HNO_3.

6.3.2 Master standard, 1000 μg Pb/ml in HNO_3/HCl. Prepare as in 6.3.1 except use the HNO_3/HCl solution in 6.2.7.

Store standards in a polyethylene bottle. Commercially available certified lead standard solutions may also be used.

7. *Procedure.*

7.1 Sampling. Collect samples for 24 hours using the procedure described in reference *10* with glass-fiber filters meeting the specifications in 6.1.1. Transport collected samples to the laboratory taking care to minimize contamination and loss of sample. (*17*).

7.2 Sample preparation.

7.2.1 Hot extraction procedure.

7.2.1.1 Cut a ¾" x 8" strip from the exposed filter using a template and a pizza cutter as described in figures 1 and 2. Other cutting procedures may be used.

Lead in ambient particulate matter collected on glass fiber filters has been shown to be uniformly distributed across the filter (*1, 3, 11*) suggesting that the position of the strip is unimportant. However, another study (*12*) has shown that when sampling near a road-way lead is not uniformly distributed across the filter. The nonuniformity has been attributed to large variations in particle size. (*16*) Therefore, when sampling near a road-way, additional strips at different positions within the filter should be analyzed.

7.2.1.2 Fold the strip in half twice and place in a 150-ml beaker. Add 15 ml of 3 *M* HNO_3 to cover the sample. The acid should completely cover the sample. Cover the beaker with a watch glass.

7.2.1.3 Place beaker on the hot-plate, contained in a fume hood, and boil gently for 30 min. Do not let the sample evaporate to dryness. *Caution:* Nitric acid fumes are toxic.

7.2.1.4 Remove beaker from hot plate and cool to near room temperature.

7.2.1.5 Quantitatively transfer the sample as follows:

7.2.1.5.1 Rinse watch glass and sides of beaker with D.I. water.

7.2.1.5.2 Decant extract and rinsings into a 100-ml volumetric flask.

7.2.1.5.3 Add D.I. water to 40 ml mark on beaker, cover with watch glass, and set aside for a minimum of 30 minutes. This is a critical step and cannot be omitted since it allows the HNO_3 trapped in the filter to diffuse into the rinse water.

7.2.1.5.4 Decant the water from the filter into the volumetric flask.

7.2.1.5.5 Rinse filter and beaker twice with D.I. water and add rinsings to volumetric flask until total volume is 80 to 85 ml.

7.2.1.5.6 Stopper flask and shake vigorously. Set aside for approximately 5 minutes or until foam has dissipated.

7.2.1.5.7 Bring solution to volume with D.I. water. Mix thoroughly.

7.2.1.5.8 Allow solution to settle for one hour before proceeding with analysis.

7.2.1.5.9 If sample is to be stored for subsequent analysis, transfer to a linear polyethylene bottle.

7.2.2 Ultrasonic extraction procedure.

7.2.2.1 Cut a ¾" x 8" strip from the exposed filter as described in section 7.2.1.1.

7.2.2.2 Fold the strip in half twice and place in a 30 ml beaker. Add 15 ml of the HNO_3/HCl solution in 6.2.6. The acid should completely cover the sample. Cover the beaker with parafilm.

The parafilm should be placed over the beaker such that none of the parafilm is in contact with water in the ultrasonic bath. Otherwise, rinsing of the parafilm (section 7.2.2.4.1) may contaminate the sample.

7.2.2.3 Place the beaker in the ultrasonication bath and operate for 30 minutes.

7.2.2.4 Quantitatively transfer the sample as follows:

7.2.2.4.1 Rinse parafilm and sides of beaker with D.I. water.

7.2.2.4.2 Decant extract and rinsings into a 100 ml volumetric flask.

7.2.2.4.3 Add 20 ml D.I. water to cover the filter strip, cover with parafilm, and set aside for a minimum of 30 minutes. This is a critical step and cannot be omitted. The sample is then processed as in sections 7.2.1.5.4 through 7.2.1.5.9.

NOTE.—Samples prepared by the hot extraction procedure are now in 0.45 *M* HNO_3. Samples prepared by the ultrasonication procedure are in 0.40 *M* HNO_3 + X *M* HCl.

8. *Analysis.*

8.1 Set the wavelength of the monochromator at 283.3 or 217.0 nm. Set or align other instrumental operating conditions as recommended by the manufacturer.

8.2 The sample can be analyzed directly from the volumetric flask, or an appropriate amount of sample decanted into a sample analysis tube. In either case, care should be taken not to disturb the settled solids.

8.3 Aspirate samples, calibration standards and blanks (section 9.2) into the flame and record the equilibrium absorbance.

8.4 Determine the lead concentration in μg Pb/ml, from the calibration curve, section 9.3.

8.5 Samples that exceed the linear calibration range should be diluted with acid of the same concentration as the calibration standards and reanalyzed.

9. *Calibration.*

9.1 Working standard, 20 μg Pb/ml. Prepared by diluting 2.0 ml of the master standard (6.3.1 if the hot acid extraction was used or 6.3.2 if the ultrasonic extraction procedure was used) to 100 ml with acid of the same concentration as used in preparing the master standard.

9.2 Calibration standards. Prepare daily by diluting the working standard, with the same acid matrix, as indicated below. Other lead concentrations may be used.

Volume of 20 μg/ml working standard, ml	Final volume, ml	Concentration μg Pb/ml
0	100	0
1.0	200	0.1
2.0	200	0.2
2.0	100	0.4
4.0	100	0.8
8.0	100	1.6
15.0	100	3.0
30.0	100	6.0
50.0	100	10.0
100.0	100	20.0

9.3 Preparation of calibration curve. Since the working range of analysis will vary depending on which lead line is used and the type of instrument, no one set of instructions for preparation of a calibration curve can be given. Select standards (plus the reagent blank), in the same acid concentration as the samples, to cover the linear absorption range indicated by the instrument manufacturer. Measure the absorbance of the blank and standards as in section 8.0. Repeat until good agreement is obtained between replicates. Plot absorbance (y-axis) versus concentration in μg Pb/ml (x-axis). Draw (or compute) a straight line through the linear portion of the curve. Do not force the calibration curve through zero. Other calibration procedures may be used.

To determine stability of the calibration curve, remeasure—alternately—one of the following calibration standards for every 10th sample analyzed: concentration ≦ 1μg Pb/ml; concentration ≦ 10 μg Pb/ml. If either standard deviates by more than 5 percent from the value predicted by the calibration curve, recalibrate and repeat the previous 10 analyses.

10. *Calculation.*

10.1 Measured air volume. Calculate the measured air volume as

$$V_m = \frac{Q_i + Q_f}{2} \times T$$

where:

V_m = Air volume sampled (uncorrected), m^3.
Q_i = Initial air flow rate, m^3/min.
Q_f = Final air flow rate, m^3/min.
T = Sampling time, min.

The flow rates Q_i and Q_f should be corrected to the temperature and pressure conditions existing at the time of orifice calibration as directed in addendum B of reference *10*, before calculation V_m.

10.2 Air volume at STP. The measured air volume is corrected to reference conditions of 760 mm Hg and 25° C as follows. The units are standard cubic meters, sm^3.

$$V_{STP} = V_m \times \frac{P_2 \times T_1}{P_1 \times T_2}$$

V_{STP} = Sample volume, sm^3, at 760 mm Hg and 298° K.
V_m = Measured volume from 10.1.
P_2 = Atmospheric pressure at time of orifice calibration, mm Hg.
P_1 = 760 mm Hg.
T_2 = Atmospheric temperature at time of orifice calibration, °K.
T_1 = 298° K.

10.3 Lead concentration. Calculate lead concentration in the air sample.

$$C = \frac{(\mu g\ Pb/ml \times 100\ ml/strip \times 12\ strips/filter) - F_b}{V_{STP}}$$

where:

C = Concentration, μg Pb/sm³.

μg Pb/ml = Lead concentration determined from section 8.

100 ml/strip = Total sample volume.

12 strips/filter = Useable filter area, 7" × 9"/Exposed area of one strip, ¾" × 7".

F_b = Lead concentration of blank filter, μg, from section 6.1.1.2.3.

V_{STP} = Air volume from 10.2.

11. *Quality control.*

¾" × 8" glass fiber filter strips containing 80 to 2000 μg Pb/strip (as lead salts) and blank strips with zero Pb content should be used to determine if the method—as being used—has any bias. Quality control charts should be established to monitor differences between measured and true values. The frequency of such checks will depend on the local quality control program.

To minimize the possibility of generating unreliable data, the user should follow practices established for assuring the quality of air pollution data, (*13*) and take part in EPA's semiannual audit program for lead analyses.

12. *Trouble shooting.*

1. During extraction of lead by the hot extraction procedure, it is important to keep the sample covered so that corrosion products—formed on fume hood surfaces which may contain lead—are not deposited in the extract.

2. The sample acid concentration should minimize corrosion of the nebulizer. However, different nebulizers may require lower acid concentrations. Lower concentrations can be used provided samples and standards have the same acid concentration.

3. Ashing of particulate samples has been found, by EPA and contractor laboratories, to be unnecessary in lead analyses by atomic absorption. Therefore, this step was omitted from the method.

4. Filtration of extracted samples, to remove particulate matter, was specifically excluded from sample preparation, because some analysts have observed losses of lead due to filtration.

5. If suspended solids should clog the nebulizer during analysis of samples, centrifuge the sample to remove the solids.

13. *Authority.*

(Secs. 109 and 301(a), Clean Air Act as amended, (42 U.S.C. 7409, 7601(a)).)

14. *References.*

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[FR Doc. 78-28050 Filed 10-4-78; 8:45 am]

APPENDIX D

APPROVAL OF SPECIFIED VARIATIONS TO EPA METHOD 5

(Letter: Byrne to Gordon, with attachment)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION VIII
1860 LINCOLN STREET
DENVER, COLORADO 80295

August 16, 1979

Ref: 8S-S

Mr. Robert J. Gordon
Pacific Environmental Services, Inc.
1930 14th Street
Santa Monica, California 90404

Dear Mr. Gordon:

The variations proposed by your organization for the source testing to be done at the ASARCO Smelter in Helena, Montana, have been reviewed. Exception (1) regarding use of Nutech Corp. impinger assembly is approved. Exception (2) regarding use of the Teflon tubing between the probe assembly and the filter box is approved with the following conditions:

a. Teflon line is heated to maintain a temperature of $120^{o} \pm 14^{o}C$ ($248^{o} \pm 25^{o}F$).

b. Device to monitor this temperature is provided. Number of monitor points dependent on the length of tubing required.

The EPA method for simultaneously determing particulate and lead emissions is enclosed for your reference. As you can see, with slight modifications described in paragraph 9.1, it is basically a Method 5 determination.

This EPA method with the above exceptions will constitute the testing methods to be employed at ASARCO.

Do not hesitate to call me if you have any further questions.

Sincerely,

Martin J. Byrne
Air Surveillance Section
Surveillance Branch
Surveillance & Analysis Division

Enclosure

PROCEDURE FOR DETERMINING THE INORGANIC LEAD EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. Particulate and gaseous lead emissions are withdrawn isokinetically from the source. The collected samples are digested in acid solution and analyzed by atomic absorption spectrometry using an air acetylene flame.

1.2 Applicability. This method is applicable for the determination of inorganic lead emissions from stationary sources.

2. Range, Sensitivity, Precision, and Interferences

2.1 Range. The upper limit can be considerably extended by dilution. For a minimum analysis accuracy of $\pm$ 10%, a minimum lead mass of 100 µg should be collected.

2.2 Analytical Sensitivity. Typical sensitivity for a 1% change in absorption (0.0044 absorbance units) are 0.2 and 0.5 µg Pb ml^{-1} for the 217.0 and 283.3 nm lines, respectively.

2.3 Precision. The within-laboratory precision, as measured by the coefficient of variation, was determined at a gray iron foundry, a lead storage battery manufacturing plant, a secondary lead smelter, and a lead recovery furnace at an alkyl lead manufacturing plant. The concentrations encountered during these tests ranged from 0.61 to 123.3 mg Pb m^{-3}. The coefficient of variation for each run, which is the standard deviation of the run expressed as a percentage of the run mean concentration, ranged from 0.2 to 9.5%.

2.4 Interferences. Sample matrix effects may interfere with the analysis for lead by flame atomic absorption. If the analyst suspects that the sample matrix is causing erroneous results, the presence of these matrix effects can be confirmed and frequently corrected for by carrying out the analysis using the Method of Standard Additions.

High concentrations of copper may interfere with the analysis of lead at 217.0 nm. This interference can be avoided by analyzing the samples for lead using the 283.3 nm lead line.

3. Apparatus

3.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure A-1. Complete construction details are given in APTD-0581; commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure A-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

3.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be $\leq 30^{o}$, and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

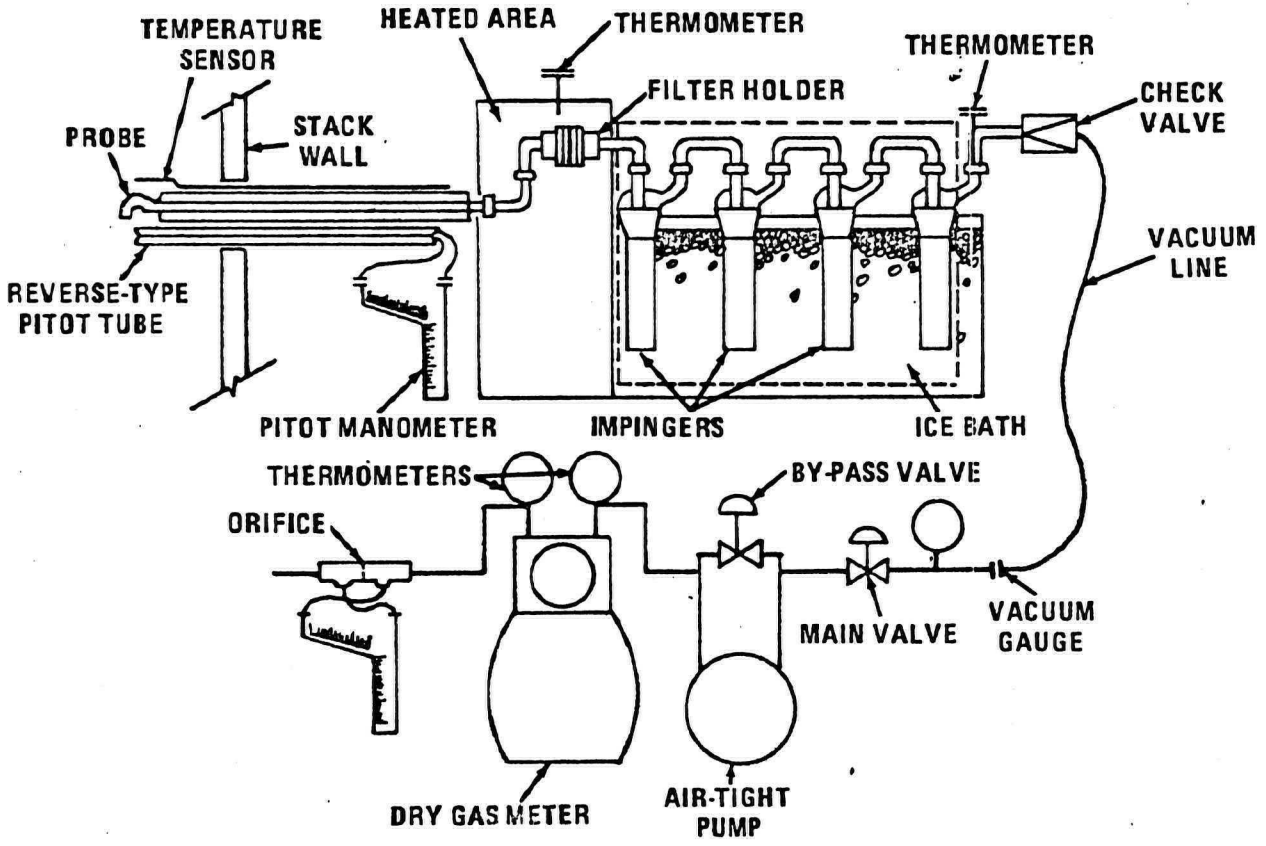

Figure A-1. Inorganic lead sampling train.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in.), or larger if higher volume sampling trains are used, inside diameter (I.D.) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be identified and calibrated (see Section 5.2).

3.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120° ± 14°C (248° ± 25°F); note that lower exit temperatures are acceptable, provided that they exceed the stack gas dew point. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480°C (900°F); quartz liners shall be used for temperatures between 480° and 900°C (900° and 1650°F). Both types of liners may be used at temperatures higher than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820°C (1508°F), and for quartz it is 1500°C (2732°F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825*, or other corrosion resistant metals) made of stainless tubing be used, subject to the approval of the Administrator.

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*Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

3.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, 40 CFR 60 Appendix A, or other device approved by the Administrator. The Pitot tube shall be attached to the probe (as shown in Figure A-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the Pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S Pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

3.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

3.1.5 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120° $\pm$ 14°C (248° $\pm$ 25°F), or such other temperature as specified by an applicable subpart the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperatur to within 3°C (5.4°F) shall be installed so that the temperature around the fil holder can be regulated and monitored during sampling. Heating systems other t the one shown in APTD-0581 may be used.

3.1.6 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be attached immediately at the outlet of the probe.

3.1.7 Impingers. Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. The first, third, and fourth impingers shall be of Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) I.D. glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall contain known quantities of 0.1 N HNO_3 (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel or equivalent desiccant. A thermometer, capable of measuring temperature to within 1°C (2°F), shall be placed at the outlet of the fourth impinger for monitoring purposes.

3.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2%, and related equipment, as shown in Figure A-1. Other metering systems capable of maintaining sampling rates within 10% of isokinetic and of determining sample volumes to within 2% may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a Pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for flow rates higher than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

3.1.9 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) shall be requested, and an adjustment for elevation differences between

the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

3.1.10 Gas Density Determination Equipment. Temperature sensor an pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3 (40 CFR 60 Appendix A). The temperature sensor shall, preferably, be permanently attached to the Pitot tul or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any me Alternatively, the sensor may be attached just prior to use in the field. No however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S Pitot tube openings (see Method 2, Figure 2-7). As a second alternative, pro that a difference of not more than 1% in the average velocity measurement is introduced, the temperature gauge need not be attached to the probe or Pit tube. (This alternative is subject to the approval of the Administrator.)

3.2 Sample Recovery. The following items are needed:

3.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out t probe liner and nozzle.

3.2.2 Glass Wash Bottles--Two.

3.2.3 Glass Sample Storage Containers. Chemically resistant, boro-silicate glass bottles, for 0.1 N HNO_3 impinger and probe solutions and wash 1000 ml. Screw cap liners shall be either rubber-backed Teflon or constructe

so as to be leak-free and resistant to chemical attack by 0.1 N HNO_3. (Narrow mouth glass bottles have been found to be less prone to leakage.)

3.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

3.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 2 ml or 1 g. The graduated cylinder shall have a minimum capacity of 500 ml, and subdivisions no greater than 5 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less.

3.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

3.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

3.2.8 Funnel. Glass, to aid in sample recovery.

3.3 Analysis.

3.3.1 Atomic Absorption Spectrophotometer. With lead hollow cathode lamp and burner for air/acetylene flame.

3.3.2 Hot Plate.

3.3.3 Erlenmeyer Flasks. 125 ml 24/40 S.

3.3.4 Membrane Filters. Millipore SCWPO 4700 or equivalent.

3.3.5 Filtering Apparatus. Millipore vacuum filtration unit, or equivalent, for use with the above membrane filter.

3.3.6 Volumetric Flasks. 100 ml, 250 ml.

4. Reagents

4.1 Sampling.

4.1.1 Filters. High purity glass fiber filters, without organic binder, exhibiting at least 99.95% efficiency ($\leq$ 0.05% penetration) on 0.3

micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpo Filters shall be Gelman Spectro Grade, or equivalent, with lot assay for Pb. Reeve Angel 934 AH and MSA 1106 BH filters have been found to be equivalent.

4.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175°C (350°F) for 2-hr. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

4.1.3 Nitric Acid, 0.1 N. Prepared from reagent grade HNO_3 and deionized, distilled water (Reagent 4.4.1, below). It may be desirable to run blanks prior to field use to eliminate a high blank on test samples. Prep by diluting 6.5 ml of concentrated HNO_3 (69%) to 1 liter with deionized, distilled water.

4.1.4 Crushed Ice.

4.1.5 Stopcock Grease. HNO_3 insoluble, heat stable, silicone greas This is not necessary if screw-on connectors with Teflon sleeves, or similar, used. Alternatively, other types of stopcock grease may be used, subject to tl approval of the Administrator.

4.2 Pretest Preparation.

4.2.1 Nitric Acid, 6 N. Prepared from reagent grade HNO_3 and deion distilled water. Prepare by diluting 390 ml of concentrated HNO_3 (69%) to 1 liter with deionized, distilled water.

4.3 Sample Recovery.

4.3.1 Nitric Acid, 0.1 N. Same as 4.1.3 above.

4.4 Analysis.

4.4.1 Water. Deionized, distilled to conform to ASTM Specification D 1193-74, Type 3.

4.4.2 Nitric Acid. Concentrated ACS reagent grade, or equivalent.

4.4.3 Nitric Acid, 50% (V/V). Dilute 500 ml of concentrated HNO_3 to 1 liter with deionized, distilled water.

4.4.4 Stock Lead Standard Solution (1000 µg Pb ml^{-1}). Dissolve 0.1598 g of reagent grade Pb $(NO_3)_2$ in about 60 ml of deionized distilled water, add 2 ml concentrated HNO_3, and dilute to 100 ml with dionized, distilled water.

4.4.5 Lead Standards.

4.4.5.1 Solution Sample Standards. Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml aliquots of the stock lead standard solution (Reagent 4.4.4) into 250 ml volumetric flasks. Add 5 ml concentrated HNO_3 to each flask and dilute to volume with deionized, distilled water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 µg Pb ml^{-1}, respectively. Additional standards at other concentrations should be prepared in a similar manner as needed.

4.4.6 Air. Of a quality suitable for atomic absorption analysis.

4.4.7 Acetylene. Of a quality suitable for atomic absorption analysis.

4.4.8 Hydrogen peroxide. ACS reagent grade or equivalent, 3% by volume.

5. Procedure

5.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

5.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless

otherwise specified herein. In addition, prior to testing, all sample-exposed surfaces shall be rinsed, first with 6 N HNO_3 then with deionized, distilled water.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and analysis.

5.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Reference Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Reference Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Reference Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight as described in Reference Method 3.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Insure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time such that (1) the sampling time per point is not less than 2 min. (or greater time interval as specified by the Administrator), and (2) a minimum lead mass of 100 µg is collected in the sample. The sampling time and volume will therefore vary from source-to-source.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

5.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of 0.1 HNO_3 in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 µg of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to insure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using tweezers or clean disposable surgical gloves, place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed, so as to prevent the sample gas stream from circumventing the

filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C (500°F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure A-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease.

Place crushed ice around the impingers.

5.1.4 Leak-Check Procedures.

5.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperature. Allow time for the temperature to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during

the leak-check. Instead, leak-check the train by first plugging the inlet to the filter and pulling a 380 mm Hg (15 in. Hg) vacuum (see note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage in excess of 4% of the average sampling rate or 0.00057 m^3/min (0.02 ft^3 min^{-1}), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause 0.1 N HNO_3 to back up into the filter. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe and immediately turn off the vacuum pump. This prevents the 0.1N HNO_3 in the impingers from being forced backward and silica gel from being entrained backward.

5.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 5.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 ft^3 min^{-1}) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction will need

to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of Reference Method 5, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 5.1.4.1 above shall be used.

5.1.4.3 Posttest Leak-Check. A leak-check is mandatory at the conclusio of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 5.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 ft^3) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. However, if a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of Method 5, or shall void the sampling run.

5.1.5 Sampling Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10% of true isokinetic unless otherwise specified by the Administrator).

For each run, record the data required on a data sheet such as the one shown in EPA Method 5, Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak-check, and when sampling is halted. Take other readings required by Figure 5-2 of Method 5 at least once at each sample point during each time

increment and additional readings when significant changes (20% variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe are at proper temperature, and that the Pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S Pitot tube coefficient is 0.85 $\pm$ 0.02, and the stack gas equivalent density (dry molecular weight) is equal to 29 $\pm$ 4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (Shigehara, 1974) are taken to compensate for the deviations.

When the stack is under significant negative pressure ($\geq$ a water column the height of the impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent 0.1 N HNO_3 from backing into the filter. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent dilution of the gas stream.

Traverse the stack cross-section, as required by Reference Method 1 or as

specified by the Administrator, without bumping the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes.

During the test run, add ice and, if necessary, salt to the ice bath, t maintain a temperature of less than 20°C (68°F) at the impinger/silica gel outlet. Also, periodically check the level and zero of the manometer.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Adminis

Note that when two or more trains are used, separate analyses of the sa fractions from each train shall be performed, unless otherwise specified by Administrator. Consult with the Administrator for details concerning the ca lation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve, remove t probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 5.1 Also, leak-check the Pitot lines as described in Method 2, Section 3.1; the l must pass this leak-check in order to validate the velocity head data.

5.1.6 Calculation of Percent Isokinetic. Calculate percent isokir (see Section 6.11 of Method 5) to determine whether the run was valid or anot test run should be made. If there was difficulty in maintaining isokinetic r due to source conditions, consult with the Administrator for possible varianc on the isokinetic rates.

5.2 Sample Recovery. Proper cleanup procedure begins as soon as

probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing liquid from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap the inlet. Remove the umbilical cord from the last impinger and cap the impinger. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the 0.1N HNO_3 used for sampling and cleanup as a blank. Place 200 ml of this 0.1N HNO_3 taken directly from the bottle being used into a glass sample container labeled "0.1N HNO_3 blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. If it is necessary to fold the filter, do so such that the sample-exposed side is inside the fold. Carefully transfer to the petri dish any visible sample matter and/or filter fibers

that adhere to the filter holder gasket by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1 N HNO_3 and placing the wash into a glass or polyethylene container. Measure and record (to the nearest ml) the total amount of 0.1N HNO_3 used for each rinse. Perform the 0.1N HNO_3 rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with 0.1N HNO_3 from a wash bottle while brushing with a stainless steel, Nylon-bristle brush. Brush until the 0.1N HNO_3 rinse shows no visible particles, the make a final rinse of the inside surface with 0.1N HNO_3.

Brush and rinse with 0.1N HNO_3 the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with 0.1N HNO_3 by tilting the probe and squirting 0.1N HNO_3 into its upper end, while rotating the probe so that all inside surfaces will be rinsed with 0.1N HNO_3. Let the 0.1N HNO_3 drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container. Follow the 0.1N HNO_3 rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1N HNO_3 into the upper end of the probe as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe and catch any 0.1N HNO_3 and sample matter that is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1N HNO_3 and none remains on the probe

liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1N HNO_3 and quantitatively collect these washings in the sample container. After the brushing make a final 0.1N HNO_3 rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize loss of sample. Between sampling runs, keep brushes clean and protected from contamination.

After insuring that all joints are wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with 0.1N HNO_3. Rinse each surface three times or more, if needed, to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO_3 washings and sample matter are collected in the sample container, tighten the lid on the sample container so that 0.1N HNO_3 will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 3. Check the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer

the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 under "Analysis."

Container No. 4. Due to the large quantity of liquid involved, the impinge solutions are placed together in a separate container. However, they may be com bined with the contents of Container No. 2 at the time of analysis in order to reduce the number of analyses required. Clean each of the first three impingers and connecting glassware in the following manner:

1. Wipe the impinger ball joints free of silicone grease and cap the joint

2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.

3. Transfer the contents of the impingers to a 500 ml graduated cylinder. The outlet ball joint cap should be removed and the contents drained through thi: opening. The impinger parts (inner and outer tubes) must not be separated while transferring their contents to the cylinder.

Measure the liquid volume to within ± 1 ml. Alternatively, determine the weight of the liquid to within ± 0.5 g by using a balance. The volume or weight of liquid present, along with a notation of any color or film observed in the impinger catch, is recorded in the log. This information is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5-3).

4. Transfer the contents of the first three impingers to Container No. 4.

5. Pour approximately 30 ml of 0.1N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1N HNO_3 through the outlet arm of each impinger into the No. 4 sample container. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

6. Wipe the ball joints of the glassware connecting the impingers free

of silicone grease and rinse each piece of glassware twice with 0.1N HNO_3; this rinse is collected in Container No. 4. (Do not rinse or brush the glass-fritted filter support.)

Mark the height of the fluid level to determine whether leakage occurred during transport. Label the container to clearly identify its contents.

Note: In steps 5 and 6 above, the total amount of 0.1N HNO_3 used for rinsing must be measured and recorded.

5.3 Analysis

5.3.1 Container No. 3. This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

5.3.2 Lead Sample Preparation and Analysis

5.3.2.1 Container No. 1. Cut the filter into strips and transfer the strips and all loose particulate matter into 125-ml Ehrlenmeyer Flask. Rinse the petri dish with 10 ml of 50% nitric acid to insure a quantitative transfer and add to the flask. (Note: if the total volume required in Section 5.3.2.3 will exceed 80 ml, it will be necessary to use a 250-ml Ehrlenmeyer flask in place of the 125-ml Ehrlenmeyer flask.)

5.3.2.2. Containers No. 2 and No. 4. Combine the contents of Containers No. 2 and No. 4 and take to dryness on a hot plate. (Note: Prior to analysis, the liquid level in Containers No. 2 and/or No. 4 should be checked; confirmation as to whether or not leakage occurred during transport should be made on the analysis sheet. If a noticeable amount of leakage has occurred, either void the sample or take steps, subject to the approval of the Administrator, to correct the final results.)

5.3.2.3 Sample Extraction for Lead. Based on the approximate stack gas

particulate concentration and the total volume of stack gas sampled, estimate the total weight of sample collected. Now transfer the residue from Containers No. 2 and No. 4 to the 125-ml Ehrlenmeyer flask that contains the filter using a rubber policeman and 10 ml of 50% (V/V) HNO_3 for every 100 µg of sample collected in the train or a minimum of 30 ml of 50% HNO_3, whichever is larger.

Place the Ehrlenmeyer flask on a hot plate and heat with periodic stirring for 30 min. at a temperature just below boiling. If the sample volume falls below 15 ml, add more nitric acid. Add 10 ml of 3% H_2O_2 and continue heating for 10 min. Add 50 ml of hot (80°C) distilled deionized water and heat for 20 min. Remove flask from heat and allow to cool. Filter the sample through a Millipore membrane filter or equivalent and transfer the filtrate to a 250-ml volumetric flask. Dilute to volume using distilled, deionized water.

5.3.2.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and place each filter in a separate 125-ml Ehrlenmeyer flask. Add 15 ml of 50% (V/V) HNO_3 and treat as described in Section 5.3.2.3 (Extraction for Lead)using 10 ml of 3% H_2O_2 and 50 ml of hot, distilled, deionized water. Filter and dilute to a total volume of 100 ml using distilled, deionized water.

5.3.2.5 0.1 N Nitric Acid Blank. Take the entire 200 ml of 0.1 N HNO_3 to dryness on a steam bath, add 15 ml of 50% (V/V) HNO_3, and treat as described in Section 5.3.2.3 (Extraction of Lead) using 10 ml of 3% H_2O_2 and 50 ml of hot, distilled, deionized water. Dilute to a total volume of 100 ml using distilled, deionized water.

5.3.2.6 Lead Determination. Calibrate the spectrophotometer as describe in Section 6.1 and determine the absorbance for each source sample, the filter blank and 0.1N HNO_3 blank. Analyze each sample three times in this manner

Make appropriate dilutions, as required, to bring all sample lead concentrations into the linear absorbance range of the spectrophotometer.

If the lead concentration of a sample is at the low end of the calibration curve and high accuracy is required, the sample can be taken to dryness on a hot plate and the residue dissolved in the appropriate volume of water to bring it into the optimum range of the calibration curve.

5.3.2.7 Mandatory Check for Matrix Effects on the Lead Results. The analysis for lead by atomic absorption is sensitive to the chemical composition and to the physical properties (viscosity, pH) of the sample (matrix effects). Since the lead procedure described here will be applied to many different sources, it can be anticipated that many different sample matrices will be encountered. Thus, it is mandatory that at least one sample from each source be checked using the Method of Additions to ascertain that the chemical composition and physical properties of the sample did not cause erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual. If the results of the Method of Additions procedure on the source sample do not agree within 5% of the value obtained by the conventional atomic absorption analysis, then all samples from the source must be reanalyzed using the Method of Additions procedure.

6. Calibration

Maintain a laboratory log of all calibrations.

6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5 (40 CFR 60 Appendix A): probe nozzle (Section 5.1); Pitot tube assemble (Section 5.2); metering system

(Section 5.3); probe heater (Section 5.4); temperature gauges (Section 5.5); barometer (Section 5.7). Note that the leak-check of the metering system (Section 5.6 of Method 5) applies to this method.

6.2 Spectrophotometer. Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement is obtained between replicates. Plot the absorbance (y-axis) versus concentration in μg Pb ml^{-1} (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or at least lie closer to the origin than ± 0.003 absorbanc units, check for incorrectly prepared standards and for curvature in the calibration curve.

To determine stability of the calibration curve, run a blank and a standa after every five samples and recalibrate, as necessary.

7. Calibrations

7.1 Nomenclature.

A_s = Stack area, m^2

$(Pb)_o$ = Total μg of lead in the source samples after correcting for al dilutions.

P_{bar} = Barometric pressure at the sampling site, mm Hg.

P_s = Absolute stack gas pressure, mm Hg.

R = Rate of lead emission, g/day.

T_m = Absolute average dry gas meter temperature, K.

T_s = Absolute stack temperature, K.

v_s = Average stack gas velocity, m/sec.

V_m = Total volume of gas sample as measured by the dry gas meter, corrected for leakage, m^3.

V_{total} = Total gas sample volume (stack conditions), m^3.

Y = Dry gas meter calibration factor.

ΔH = Average pressure differential across the orifice meter, mm H_2O.

7.2 Calculate V_m, the total volume of dry gas metered (corrected for leakage, if necessary, as outlined in Section 6.3 of Method 5, 40 CFR 60, Appendix A).

7.3 Calculate the volume of water vapor and the moisture content of the stack gas, from data obtained in this testing, use Equations (5.2) and (5.3) of Method 5, 40 CFR 60, Appendix A.

7.4 Calculate, v_s, the average stack gas velocity, using Equation (2-9) of Method 2, 40 CFR 60, Appendix A; use velocity head (ΔP), temperature, pressure, and moisture data from this field test.

7.5 Calculate the total gas sample volume at stack conditions, using the following equation:

$$V_{total} = V_m Y \left[\frac{T_s}{T_m}\right]\left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_s}\right] \qquad \text{(A-1)}$$

7.6 Total Lead in Source Sample. For each source sample correct the average absorbance for the contribution of the filter blank and the 0.1 N HNO_3 blank. Use the calibration curve and this corrected absorbance to determine the lead concentration in the sample aspirated into the spectrophotometer.

Calculate the total lead content in the original source sample $(Pb)_o$; correcting for all the dilutions that were made to bring the lead concentration of the sample into the linear range of the spectrophotometer.

7.7 Total Lead Emission. Calculate the total amount of lead emitted

from each stack per day by Equation (A-2). This equation is applicable for continuous operations. For cyclic operations, use only the time per day each stack is in operation The total lead emissions from a source will be the summation of results from all stacks.

$$R = \left[\frac{(Pb)_o \; v_s \; A_s}{V_{total}}\right] \left[\frac{86400 \text{ seconds/day}}{10^6 \; \mu g/g}\right] \qquad \text{(A-2)}$$

8. Isokinetic Variation. Determine the isokinetic variation in the sampling rate using Equation (5-7) of Method 5, 40 CFR 60, Appendix A and the raw data from this testing.

8.1 Acceptable Isokinetic Results. The following range sets the limit on acceptable isokinetic sampling results:

If $90\% \leq I \leq 100\%$, the results are acceptable. If the results are low in comparison with the emission standard and I is beyond the acceptable range, or if I is less than 90%, the Administrator may opt to accept the results. Otherwise, reject the results and repeat the test.

9. Alternate Test Methods for Inorganic Lead

9.1 Simultaneous Determination of Particulate and Lead Emissions. Method 5 as described in 40 CFR 60, Appendix A, is an acceptable alternate test method provided that: (1) 0.1N HNO_3 is used in the impingers; (2) a glass fi filter with a low lead background is used; and (3) the entire train contents, including the impingers, are treated and analyzed for lead as described in Section 5 of this Test Method.

9.2 Filter located Between Third and Fourth Impinger. Location of the filter between the third and fourth impinger is an acceptable alternative

method provided that the filter is included in the analysis for lead.

9.3 In-Stack Filter. Use of an in-stack filter is an acceptable alternate method provided that: (1) the in-stack filter is followed by a glass-lined probe and at least two impingers that each contain 100 ml of 0.1N HNO_3; and (2) the probe and impinger contents are recovered and analyzed for lead.

10. Bibliography

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APPENDIX E

TYPICAL CALCULATION: MAIN STACK RUN NO. 1

certified testing laboratories, inc.

CTL - ENVIRONMENTAL SERVICES

TYPICAL CALCULATION MAIN STACK RUN #1

1) Dry Gas Volume, Standard Conditions

$$Vm\ (std) = Vm \times Y \times 17.647 \times \frac{Pbar + \Delta H/13.6}{Tm}$$

Where
- Vm (std) = Corrected gas volume
- Vm = Measured gas volume
- Y = Gas Meter Correction
- Pbar = Measured barometric pressure
- ΔH = Average pressure differential, orifice meter
- Tm = Average gas meter temperature, °R

$$Vm\ (std) = 74.285 \times 1.004 \times 17.647 \frac{26.04 + \frac{2.5}{13.6}}{69.0 + 460} = 65.24$$

2) Particulates Collected

On Filter	0.0287 g
Probe Wash and Impinger Catch	0.1794
Total	0.2081 g

3) Water Vapor

Moisture Collected:

Impinger No. 1	3.2	grams
2	2.4	
3	1.6	
4	16.3	
Total	23.5	grams

Volume of water vapor (Eng. units): Weight of moisture (grams) x 0.04715

23.5 x 0.04715 = 1.11 SCF

4) Moisture Content of Stack Gases

$$\text{Moisture Content} = \frac{1.11}{1.11 + 65.24} \times 100 = 1.7\%$$

5) Molecular Weight of Stack Gases, Dry

$$MW = (0.44 \times CO_2) + (0.32 \times O_2) + (0.28 \times N_2)$$

$$(0.44 \times 2.0) + (0.32 \times 18.6) + (0.28 \times 79.4) = 29.1$$

6) Molecular Weight of Stack Gases, Wet

$$MW = 29.1\ (1 - 0.017) + (18 \times 0.017) = 28.9$$

7) Average Stack Gas Velocity

$$Vs = 85.48 \times Cp \times (\Delta p\ avg)^{\frac{1}{2}} \times \left[\frac{Ts\ avg}{Ps \times Ms}\right]^{\frac{1}{2}}$$

Where Vs = Average velocity, ft/sec
Cp = Pitot tube coefficient
Δp avg = Velocity head, in. of water
Ts avg = Average stack temperature, ^{o}R
Ps = Abs. stack pressure, in. of Hg
Ms = Molecular weight of stack gases, wet

$$Vs = 85.48 \times 0.819 \times (0.801)^{\frac{1}{2}} \times \left[\frac{171 + 460}{25.5 \times 28.9}\right]^{\frac{1}{2}} = 57.97 \text{ ft/sec}$$

8) Isokinetic Sampling

$$\%I = 0.0945 \times \frac{Ts\ Vm\ std}{Ps\ Vs\ An\ \emptyset\ (1 - Bws)}$$

Where Ts = Stack gas temperature, ^{o}R
Vm std = Corrected sample volume
Ps = Abs. stack gas pressure
Vs = Stack gas velocity
An = Cross-sectional area of nozzle, ft^2
θ = Sampling time, min
Bws = Moisture in gas steam, vol. fraction

$$\%I = 0.0945 \times \frac{(171 + 460)\ (65.24)}{25.5 \times 57.96 \times 0.000341 \times 84 \times (1-.017)} = 93.5\ \%$$

9) Total Gas Volume at Stack Conditions

$$V_{Total} = V_m\ (std)\ Y\left(\frac{Pstd}{Tstd}\right)\left(\frac{Tstack}{Pstack}\right)$$

$$V_{Total} = 65.24\ (1.004)(.0567)\ \left(\frac{171 + 460}{25.5}\right) = 91.5 \text{ cubic feet}$$

10) Mass Emission Rate

$$R = \left(\frac{mg\ \ Metal\ Vs\ As}{V_{Total}}\right)\left(\frac{86400\ sec/day}{10^3 mg/gram}\right)$$

$$R_{Pb} = \frac{(10.8)(57.96)\ (56.19)}{91.50}\left(\frac{86400}{10^3}\right) = 33220 \text{ grams/day}$$

APPENDIX F

STACK TEST DATA SHEETS

RUN 1 MAIN STACK 8.21.79 MAIN 1 VENTURI Horiz

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Cp = .819

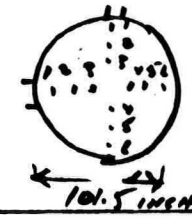
Plant Banco Hesperia Mont.
Run Number 1 - Venturi Horiz
Location Main Stack
Date ~~8-21-79~~ 9-21-79
Operator R.D
Sample case number 2
Monitor Unit number #1

CO_2 2.0%
O_2 18.6%
Mw Mw = 29.06

Run #1

Ambient temperature, °F 12°C = 54°
Barometric pressure, in. Hg 26.04
Assumed moisture, % 5
Heater box setting, °F 6
Pitobe tip diameter, in ~~.250~~ .250
Pitobe length, ft. 11FT 5.5.
Pitobe heater setting 6

STACK Pressure - 25.70" Hg
STACK DIAM 101.5 INCHES



Cp - .819
K ~~3.4~~ 3.1

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F 270°	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	8:26	435.002	.60	1.82	1.82		48	2.5"	~~44~~	44	25.70	167
2	8 33	440 3	.88	2.7	2.7		52°	4.0	250	39	"	170
3	8:40	447.6	.94	2.85	2.85		58	4.0	252	42	"	165
4	8:47		1.05	3.20	3.20		64	4.5	255	44	"	165
5	8:54	460 7	1.05	3.20	3.20		69°	4.5	250	43	"	165
6	9:01	467.5	.60	1.82	1.82		74	2.5	250	44	"	165
STOP	9:08	472.517					75					
1	9:56	472.661	.56	1.72			71	3.0	250	48	"	165
2	10:03	479	.70	2.1			73	5.0	250	42	"	180
3	10:10	[illegible]	.76	2.3			75	6.0	255	45	"	180
4	10:17	490	.93	2.8			79	6.5	260	47	"	175
5	10:24	496	.95	2.9			83	6.5	260	48	"	175
6	10:31	502	.75	2.25			87	6.0	260	44	"	175
STOP	10 38	509.287	0.895	Gas AVG		7	69				X	171
	Δ	74.285		Y = 2.5								

IMPINGER wt. before wt. after Δ

	wt. before	wt. after	Δ
#1	[illegible]	534.6	3.2
#2	528.1	531.3	2.4
#3	427.5	429.1	1.6
#4	682.5	698.8	16.3
			23.5 grams

FILTER before after

	before	after
#10	0.4037	0.4324
#12		
#13		
#14		

Δ LEAK CHECK 0 - 12 - #2
28.7 Pre <.01 CFM @ 15 PSI
Post <.01 CFM @ 14 PSI

0030269

ASARCO Helena, Montana

Plant MAIN STACK
Run Number 2
Location ~~Before Bag Stack~~ MAIN STACK
Date 9-26-79
Operator [illegible]
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard

South Gate, CA 90280

V_{STD} 46.107 SCF
V_{STOC} 58.102 ft/sec
% H_2O 3%
% ISO 94.3%
Grain load 0.009 gr/dscf

MAIN STACK Run 2
Ambient temperature, °F 70°
Barometric pressure, in. Hg ~~25.80~~ 25.85
Assumed moisture, % 5
Heater box setting, °F 250
Pitobe tip diameter, in. .251
Pitobe length, ft. 11 S.S.
Pitobe heater setting Hi
STACK P = 25.52

94.6

Point	Clock Time (5 min per point; 0-60 min)	Dry Gas Meter ft³ (Δ=54.438)	Pitot, in. H_2O ΔP ($(\sqrt{\Delta p})_{av}$=.891)	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F	Dry gas temperature, °F	Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F (250)	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	12:30	510.694	.58	1.82			74	5"	~~[illegible]~~	53	25.52	170
2	12:35	514	.68	2.15			75	5"	255	41	"	175
3	12:40	519	.76	2.4			78	6"	265	50	"	175
4	12:45	523	.92	2.9			81	6.5"	265	52		
5	12:50		.85	2.4			85	"	265	52		175
6	12:55	533	.74	2.35			88	"	260	52		175
STOP	1:00	537.000										
1	1:46	537.183	.57	1.75			85	6.5	250	58		160
2	1:51/1:55	540	.95	2.9			85	"	270	60		180
3	~~2:37~~ 2:43	546	1.00	3.2			87	"	260	38		180
4	2:48	551	1.00	3.2			90	"	265	44		180
5	2:53	555	.96	2.9			93	"	250	46		180
6	~~2:58~~ 3:01	561	.62	2.0			94	"	250	48		185
STOP	3:06 pm	565.132	0.891	2.5			84.6					175.8
STOP	5:30 sec into run											
Restart at 1:55												
Restart at 2:33:30												

Left margin: Horiz. (points 1–6, first traverse); VERT (points 1–6, second traverse); * 2:33:30 (point 2)

OFF AT 2:35:30 RESTART 2:40:30 * STOP 2:57 (AMOUNT 60) RESTART 3:00

IMPINGER	wt. before	wt. after	
#1	539.55	547.0	Δ=7.45
#2	538.87	546.9	8.05
#3	~~538.85~~ 429.6	431.4	1.8
#4	660.9	675.0	14.1

FILTER	before	after
#1 11	.4073	0.4347
#2		
#3		
#4		

Δ LEAK CHECK

27.4 in. Hg Pre 0 CFM @ 12 PSIG

Post 0.01 CFM @ 12 PSIG

[signature]

0030270

Plant ASARCO
Run Number 3
Location Main STACK
Date 9-21-79
Operator [illegible]
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Ambient temperature, °F 29°C
Barometric pressure, in. Hg ~~29.8~~ 25.85
Assumed moisture, % ~~5~~ 3
Heater box setting, °F 250
Pitobe tip diameter, in. .250
Pitobe length, ft. 11 S.S.
Pitobe heater setting Hi

%150-9M

0-10 min

Point	Clock Time (5 min. per point)	Dry Gas Meter ft³	Pitot, in. H_2O ΔP ($\sqrt{\Delta P}$ = .856)	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F	Dry gas temperature, °F	Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
VERT. 1	4:46	565.793	.53	1.65			97	4"	252	56	25.5	180
2	4:51		.85	2.6			99	6	251	45		190
3	4:56	574.5	.90	2.8			100	"	250	45		185
4	5:01	579.0	1.00	3.0			103	7	260	54		180
5	5:06	584	.95	2.9			105	6	265	52		185
6	5:11	589	.60	1.8			107	5	265	52		180
STOP	5:16	593.780										
HORIZ. 1	5:58	593.928	.53	1.65			102	4	250	53	25.5	170
2	6:03	597	.67	2.1			102	5	275	43		185
3	6:08	601	~~.[illegible]~~ .90	~~2.[illegible]~~ 2.8			103	5.T	260	48		185
4	6:13		.86	2.7			104	6	255	48		180
5	6:18		.72	2.2			105	6	255	52		180
6	6:23	615.7	.42	1.3			106		252	54		180
				$\bar{x}$ 2.3								$\bar{x}$ = 181.7
STOP	6:28	619.196				$\bar{x}$	102.7					
		Δ 53.403	.856 = $\sqrt{\Delta P}$									

IMPINGER	wt. before	wt. after	Δ
#1		~~541.3~~ 547.5	18.4
#2	[illegible]	~~541.5~~ 541.3	9.9
#3	[illegible]	429.3	1.7
#4	[illegible]	695.2	12.2
			42.2 g H_2O

4 11 hr probe [illegible]

FILTER	before	after	Δ
#1 12	.4125	0.4336	21.1 mg
~~#2~~			
~~#3~~			
~~#4~~			

LEAK CHECK <0.01 12

Pre <0.01 CFM @ 12 PSIG

Post <0.01 CFM @ 12 PSIG

National Enforcement EPA Sept 21, 1979

0030271

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard

South Gate, CA 90280

Plant ASARCO - Helena

Run Number 1

Location ZINC STACK

Date 9-22-79

Operator RGD

Sample case number # 2

Monitor Unit number # 1

ZINC STACK #1

Ambient temperature, °F 65

Barometric pressure, in. Hg 25.92

Assumed moisture, % 5%

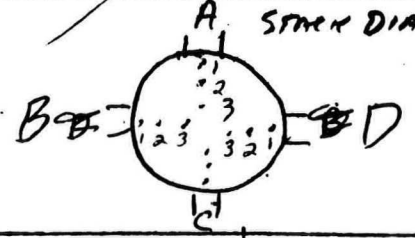
Heater box setting, °F 250

Pitobe tip diameter, in. .253

Pitobe length, ft. 5

Pitobe heater setting Hi

STACK DIAM 11'3"



3 PORTS IN AT START

7 IN TOTAL

TEFLON FLEX LINER (180°F)

$C_p = .853$ .833

K = 3.1

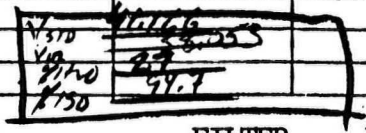
$\gamma = 1.004$

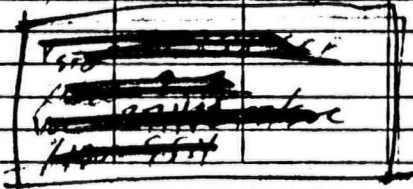
O_2 - 0.4%

O_2 - 20.6%

Moisture 28.8%

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
		619.790	(ΔP)avg .556									
1A	3:29 PM		0.25	.78		78		3	210	43	25.08	200
B	3:56	623.5	0.38	1.20		79		4	210	42		210
C	5:43	628	0.41	1.30		80		4	225	44		215
2A	3:54	632.505	0.25	.78		82		4	210	50		220
B	4:01	637	0.32	1.00		87		4	225	50		220
C	4:05	640	0.32	1.00		92		4	270	52		220
3A	4:31	644.335	0.23	0.72		93		4	230	46		220
B	4:38	648.86	0.33	1.04		93		4	250	45		220
C	4:45	653	0.33	1.04		93		4	245	45		240
1	5:02	656.719	0.22	0.70		95		3	225	54		230
2	5:09	660	0.35	1.10		98		4	230	47		240
C	5:16	664.4	0.35	1.10		100		4	245	49		250
Stop	5:23	668.865										
				X = 0.98		X = 89						X = 223.7
	Δ	40.865	(ΔP)avg .556									
Time	= 84 min											





IMPINGER	wt. before	wt. after	Δ
#1	533.6	541.5	7.9
#2	532.6	536.8	4.2
#3	427.4	430.4	3.0
#4	649.4	658.6	9.2
		TOTAL	24.3 Grams H_2O
#4	660.0	671.2	11.2

FILTER	before	after	Δ
#1 13	.4056	0.4118	6.2mg
#2			
#3			
#4			

0.002 GR/DSCF

LEAK CHECK

Pre <0.01 CFM @ 14 PSIG "Hg

Post <0.01 CFM @ 18 PSIG "Hg

Norton J. Byrne EPA 422 M

Norton J. Byrne

$f_{.150} = 96.4$

00030272

ZINC STACK RUN 2

Plant ASARCO - HAYDEN MINE
Run Number 2
Location ZINC STACK
Date 9/23
Operator KD
Sample case number 212
Monitor Unit number #1

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

$V_{m(std)}$ ~~[illegible] SCF~~ 44.049 SCF
$\%H_2O$ ~~[illegible]~~ 1.3%
VELOCITY ~~[illegible] FT/SEC~~ 35.099 FT/SEC
%ISO ~~[illegible]~~ 95.1
GRAIN LOAD 0.002 GR/DSCF

Ambient temperature, °F 46
Barometric pressure, in. Hg 26.00
Assumed moisture, % 3
Heater box setting, °F H
Pitobe tip diameter, in. 0.253
Pitobe length, ft. GLASS SET-IN-A-TUBE
Pitobe heater setting H
TEFLON FLEX LINE TEMP 150

%ISO = 96.4

γ = 1.004 C_p = .833

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	8 [illegible]	667.285	[illegible].33	1.03			53	4"	225	39	25.94	210
2	8 22		.40	1.25			55	4	260	37		210
3	8.29	678	.44	1.42			58	4	255	38		210
EMISSION	8 36	682.806	[illegible]	[illegible]			—	—	—	—		[illegible]
1	8 44		.30	.98			59	3	225	42		200
South 2	8 51	687	.34	1.05			60	4	240	41		210
3	8.58		.34	1.05			61		260	42		210
[illegible]	9 04	694.686	—	—			—	—	—	—		—
1	9 21		.23	.70			63	3	250	48		210
2	9 28	698 3	.35	1.15			64	3	250	42		210
3	[illegible]9.35		.35	1.15			65	4	225	41		210
STOP	9 42	706.624	—	—			—	—	—	—		—
South 1	9.48		.26	.85			64	3	260	47		200
2	9.55	711	.35	1.15			64	3	255	43		220
3	10.02	714 7	.36	1.18			64	3	245	43		230
STOP	10 09	718.933	—	—			—	—	—	—		210.8
Total	81 min	Δ=49.648	$(\Delta p)_{avg}$ = .579	X=1.08		X=60.8					X	

IMPINGER	wt. before	wt. after	Δ
#1	520.2	524.8	4.6
#2	522.0	522.3	0.3
#3	429.3	429.3	0.0
#4	686.2	693.3	7.1

FILTER	before	after
#1	0.4047	0.4094
#2		
#3		
#4	[illegible]	[illegible]

Δ LEAK CHECK
Pre <0.01 CFM @ 15 ~~PSIG~~ "Hg
Post <0.01 CFM @ 15 ~~PSIG~~ "Hg

0030273

Zinc Stack Run 3

CO_2 1.8 1.8
O_2 18.4 18.4

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant ASARCO - Hayden Mont
Run Number 3
Location Zinc Stack
Date 5/23/79
Operator ASD
Sample case number 1
Monitor Unit number 1

V_{std} = 43.687 SCF
$V_{velocity}$ = 35.945 FPS
$\%H_2O$ = 1.9%
% I50 = 99.8%
Grain load 0.0026 GR/DSCF

% I50 = 96.5

Ambient temperature, °F 52
Barometric pressure, in. Hg 26.00
Assumed moisture, % 3
Heater box setting, °F Hi
Pitobe tip diameter, in. .253
Pitobe length, ft. 5 FT Glass + 12 Ft Tygon
Pitobe heater setting Hi
Tygon Temp 250
C_p = .833

Point	Clock Time 10:59	Dry Gas Meter ft³	Pitot, in. H_2O ΔP ($\sqrt{\Delta P}$ = .585)	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	~~11:00~~	719.266	.34	1.1			65	3	225	55	25.94	200
2	11:06		.44	1.44			68	3	260	48		220
3	11:13	727.8	.42	1.4			70	4	250	49		220
1	11:26		.26	.84			72		225	59		220
2	11:33		.33	1.1			73	3	260	48		210
3	11:40	744.728 stop	.35	1.15			75	3	248	49		220
1	11:47		.25	.82			75	2	240	66		230
2	1202		.35	1.15			76	2	240	50		230
3	12:09		.37	1.2			78		240	54		240
1	12:23	752.175	.24	.78			77	2	240	55		220
2	12:30	760.8	.40 ~~.39~~	~~1.3~~ 1.3			78	3	235	56		240
3	12:37		.39	1.3			79	3	235	57		240
	12:44	769.728	$\sqrt{\Delta P}$=0.585	1.13			73.8					TF= 224.2
		Δ= 50.462										
	Time 84 min											

IMPINGER	wt. before	wt. after	
#1	524.8	528.0	3.2
#2	542.4	543.7	1.3
#3	436.1	438.4	2.3
#4	660.0	671.2	11.2

FILTER	before	after
#15	0.4117	0.4165
#2		
#3		
#4		

△ LEAK CHECK
Pre <0.01 CFM @ 14 "Hg
Post <0.01 CFM @ 22 "Hg

0030274

Baghouse Stack #1 Run 3

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant ASARCO - HAYDEN MINE
Run Number 3
Location Baghouse #1
Date 9/28/79
Operator RJD
Sample case number 1
Monitor Unit number 2

$V_{m\,STD}$ ~~35.040~~ 83.740 SCF
~~SCM~~ 15.86~~6~~ ~~FT/SEC~~
V_{CR} ~~FT/SEC~~
%H.O ~~FT~~ 1.7
%150 ~~99.3%~~ ~~97.7%~~ 99.6

Ambient temperature, °F 50
Barometric pressure, in. Hg 25.94
Assumed moisture, %
Heater box setting, °F 250
Pitobe tip diameter, in. .485
Pitobe length, ft. 11' 5S
Pitobe heater setting Hi
C_p: ~~.835~~ .819

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	8:18	555.394	.04		2.3		56	5"	230	46	25.94	140
2	8:20		.06		3.4		56					
3	8:22		.06		3.4		58	6"	240	46		160
4	8:24		.08		4.6		60		230	44		160
5	8:26		.09		5.1		62	8"	"	"		160
6	8:28	566.7	.10		5.7		66	9"	235	44		160
7	8:30		.13		7.4		70	12"	235	50		160
8	8:32	573.988	.12		6.9		70	12"	235	52		170
9	~~8:56~~	~~578.~~	.05		2.9		64		240	56		160
10	8:58		.05		2.9		68		240	56		160
11	9:00		.05		2.9		69	5	240	56		160
12	9:02		.05		2.9		70		240	58		
13	9:04		.05		2.9		71		250	56		160
14	9:06		.07		4.0		74		255	54		160
15	9:08		.10		5.7		76		250	54		170
16	9:10		.12		6.9		80		255	52		170
17	9:22		.04		2.3		76		235	56		160
18	9:24		.04		2.3		78		235	54		
19	9:26		.03		1.7		80		250			160

IMPINGER	wt. before	wt. after	Δ
#1	540.25	~~544.6~~ 539.1	-1.15
#2	539.5	549.6	10.1
#3	429.4	432.0	2.6
#4	680.8	700.1	19.3
			30.85

FILTER	before	after
#1 20	0.4032	0.4224
#2		
#3		
#4		

Δ LEAK CHECK
Pre <0.01 CFM @ 15 ~~PSIG~~ "Hg
Post <0.01 CFM @ 20 PSIG

00030275

Plant ASARCO - Helena
Run Number 3
Location Baghouse 1
Date 9-28-79
Operator [illegible]
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

page 2

Ambient temperature, °F
Barometric pressure, in. Hg
Assumed moisture, %
Heater box setting, °F
Pitobe tip diameter, in.
Pitobe length, ft.
Pitobe heater setting

	Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
	20	9:28	596.	.03		1.7		80		245	50		170
3	21	9:30		.04		2.3		82		255	52		170
	22	9:32		.06		3.4		84		255	52		170
	23	9:34		.10		5.7		86		250	54		170
	24	9:36		.12		6.8		85		255	54		
	25	9:48	606.528	.03		1.7		84		255	56		170
	26	9:50		.03		1.7		86		260	56		
	27	9:52		.03		1.7		88		260	57		170
4	28	9:54		.01		0.6		90					
	29	9:56	612.	.01		0.6		90		250	49		170
	30	9:58		.09		5.1		90		255	49		180
	31	10:00	615	.13		7.4		94		255	50		180
	32	10:02		.10		5.7		96		260	51		180
	33	10:17	620.816	.02		1.15		88		260	50		160
5	34	10:19		.03		1.7		89		255	51		160
	35	10:21		.05		2.9		89		250	52		170
	36	10:23		.05		2.9		90		250	53		170
	37	10:25		.06		3.4		92			50		180
	38	10:27		.09		5.1		94		250	50		

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre <.001 CFM @ 15" Hg PSIG
Post ____ CFM @ ____ PSIG

[illegible]

0030276

Plant Asarco - Helena
Run Number 3
Location Baghouse #1
Date 9-23-79
Operator RJO
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

page 3

Ambient temperature, °F
Barometric pressure, in. Hg 25.92 "Hg
Assumed moisture, %
Heater box setting, °F
Pitobe tip diameter, in.
Pitobe length, ft.
Pitobe heater setting

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
39	10:29	631.	.11		6.3		98	8"	255	50		180
40	10:31		.10		5.7		99		260	52		"
41	10:42	637.253	.02		1.15		92	3"	245	52		160
42	10:44		.02		1.15		94		245	53		160
43	10:46		.03		1.7		94		250	54		
44	10:48		.05		2.9		94		250	56		170
45	10:50		.10		5.7		96		255	56		170
46	10:52		.13		7.4		100		245	48		180
47	10:54		.11		6.3		102		248	48		180
48	10:56		.11		6.3		104		245	50		180
		653.953	$\sqrt{\Delta P}_{Avg}$: 0.2472		$\bar{X}$=3.8		$\bar{X}$=82.4					166
		Δ 98.559										

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

Brandon J Byrne
Alaska

0030277

Page 1 of 3

Plant ASARCO - Hayden Mont
Run Number 4
Location Stack #1
Date 9/28/79
Operator RDD
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

$V_{m\,STD}$ 74.661
$V_{s\,AVG}$ 14.326 FT/SEC
%H_2O 1.6
%ISO 100.1%

Ambient temperature, °F 50
Barometric pressure, in. Hg 25.94
Assumed moisture, % 2
Heater box setting, °F 250
Pitobe tip diameter, in. 0.485
Pitobe length, ft. 11 FT 55
Pitobe heater setting Hi

GRAIN LOADING 0.004 GR/DSCF

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP ($\sqrt{\Delta P}_{AV}$ = 0.222)	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
48	2:32	654.342	.02		1.15		80	3	230	44	25.94	160
47	2:34	655.4	.005 .08		4.6		82		230	44		160
46	2:36		.08		4.6		87		235	44		160
45	2:38		.06		3.4		84		235	49		160
44	2:40		.05		2.9		84			48		
43	2:42		.03		1.7		84		240	48		160
42	2:44		<.005		0		84		250	46		160
41	2:46		<.005		0		—		—	—		—
40	2:56	664.368	0.10		5.7		86	6.0"	255	49		170
39	2:58		0.08		4.6		85		255	46		170
38	3:00		0.08		4.6		86		255	46		170
37	3:02		0.06		3.4		92		250	48		170
36	3:04		0.04		2.3		93		250	50		170
35	3:06		0.04		2.3		94		255	50		170
34	3:08		0.03		1.7		96		250	51		180
33	3:10		0.03		1.7		96		255	53		180
32	3:20	680.576	0.10		5.7		94		245	44		180
31	3:22		0.10		5.7		94		250	44		180
30	3:24		0.09		5.1		96		255	46		180

IMPINGER	wt. before	wt. after	
#1	539.6	539.5	-.1
#2	541.9	546.8	4.9
#3	429.7	432.2	2.5
#4	728.3	747.2	18.9
			71.7

FILTER	before	after
#22	0.4122	0.4311
#2		
#3		
#4		

Δ LEAK CHECK 18.9
Pre <0.01 CFM @ 15 "Hg
Post <0.01 CFM @ 15 "Hg

Standard Engineering

0030278

Plant Asarco - Helena Mt.
Run Number Run # 4
Location Baghouse 1
Date 9-27-79
Operator RQD
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES
2905 E. Century Boulevard
South Gate, CA 90280

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Ambient temperature, °F
Barometric pressure, in. Hg
Assumed moisture, %
Heater box setting, °F
Pitobe tip diameter, in.
Pitobe length, ft.
Pitobe heater setting

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
29	3:26	688	.05		2.9		98		250	50		180
28	3:28		.05		2.9		98		250	50		180
27	3:30		.04		2.3		99		255	52		
26	3:32		.04		2.3		100		255	53		180
25	3:34		.04		2.3		100		"	"		"
24	3:50	697.706	.06		3.4		92		240	46		180
23	3:52		.06		3.4		93		245	46		190
22	3:54		.05		2.9		94		245	46		190
21	3:56		<.005	shake	0		0					
20	3:58		.04		2.3		93		250	46		190
19	4:00		.04		2.3		94		250	46		190
18	4:02		.04		2.3		96		255	44		190
17	4:04		.05		2.9		96		255	45		190
16	4:29	710.671	.07		4.0		89		240	46		190
15	4:31		.09		5.1		90		250	46		190
14	4:33		.06		3.4		90		250	50		190
13	4:35		.05		2.9		91		"	"		190
12	4:37		.04		2.3		90		"	52		190
11	4:39		.04		2.3		92					

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

0030279

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Page 3

Plant Asarco, Helena MT
Run Number 4
Location Baghouse # 1
Date 9-27-79
Operator RD
Sample case number 1
Monitor Unit number 2

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
10	4:41		.03		1.70		92		255	52		190
9	4:43		.05		2.9		92		"	"		190
8	5:17	726.308	.06		3.4		84		250	50		180
7	5:19		.08		4.6		86		255	52		180
6	5:21		.09		5.1		86		260	52		
5	5:23		.08		4.6		88		250	54		180
4	5:25		.06		3.4		90					180
3	5:27		.06		3.4		90					180
2	5:29		.05		2.9		92					180
1	5:31		.05		2.9		92					
		743.755										X 1765
		Δ=87.413	(Σ√ΔP)²=0.22		X=3.09		X 90.8					

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

0030280

Plant Asarco - Hayden Plant
Run Number 5
Location Bag House Stack #1
Date 9/25/79
Operator KJD
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES
2905 E. Century Boulevard
South Gate, CA 90280

V_{mstd} 87.563 SCF
$\%H_2O$ 1.7%
Vel_s 16.335 Ft/Sec
%I 99.6%

Ambient temperature, °F 50
Barometric pressure, in. Hg 26.07
Assumed moisture, % 5
Heater box setting, °F 256
Pitobe tip diameter, in. .485
Pitobe length, ft. 11' 5S
Pitobe heater setting 11
Meter coma Factor 0.998
C_p = .819

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	10:32	744.276	.02		1.15		60		255		26.07	130
2	10:34		.05		2.85		60		235	58		130
3	10:36		.05		2.85		62		250	54		150
4	10:38		.08		4.6		63		250	52		160
5	10:40		.10		5.7		66		250	52		160
6	10:42		.11		6.3		70	11"	250	52		160
7	10:44		.12		6.8		73	11"	250	56		160
8	10:46		.10		5.7		76		250	56		160
9	10:53	762.268	.05		2.85		75		255	52		160
10	10:55		.05		2.85		79		250	54		150
11	10:57		.05		2.85		80		"	54		"
12	10:59		.05		2.85		82		250	56		160
13	11:01		.06		3.4		84		245	56		160
14	11:03		.07		4.0		86		250	52		160
15	11:05		.08		4.6		88		255	52		
16	11:07		.09		5.1		90		255	55		160
17	11:15	779.034	.05		2.85		88		250	48		160
18	11:17		.05		2.85		91		250	50		160
19	11:19		.05		2.85		92					

IMPINGER	wt. before	wt. after	
#1	530.4	521.2	-9.2
#2	542.3	555.1	12.8
#3	429.8	435.7	5.9
#4	695.8	718.7	22.9
			32.4

FILTER	before	after	Δ
#24	0.4102	0.4254	15.2
#2			
#3			
#4			

Δ LEAK CHECK
Pre 0.01 CFM @ 15" Hg
Post 0.01 CFM @ 15" Hg

0030281

Plant Pasco - Helena
Run Number 5
Location Baghouse #1
Date 9-29-79
Operator RJD
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

page #2

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F 250
Pitobe tip diameter, in. .485
Pitobe length, ft. 11' 5S
Pitobe heater setting Hi

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
20	11:21		.05		2.85		94		250	53		160
21	11:23		.05		2.85		96		250	53		160
22	11:25		.06		3.4		98		250	55		160
23	11:27		.08		4.6		100		245	52		160
24	11:29		.09		5.1		102		245	53		160
25	11:40	796.046	.04		2.3		98		250	54		160
26	11:42		.05		2.85		98		250	54		160
27	11:44		.04		2.3		100		250	48		160
28	11:46		.05		2.85		101		250	48		170
29	11:48		.06		3.4		102		245	47		170
30	11:50		.08		4.6		103		245	48		170
31	11:52		.11		6.3		104		250	50		170
32	11:54		.12	shake	6.8		102		245	52		170
33	12:02	813.609	.02		1.15		100		240	52		170
34	12:04		.03		1.7		102		240	53		170
35	12:06		.03		1.7		102		240	54		160
36	12:08		.05		2.85		103		245	54		160
37	12:10		.08		4.6		104		250	46		160
38	12:12		.10		5.7		106		250	48		160

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

00030282

Plant Asarco - Helena, Mt
Run Number 5
Location Baghouse #1
Date 9-29-79
Operator CJD
Sample case number 1
Monitor Unit number 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

page 3

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % 5
Heater box setting, °F 250
Pitobe tip diameter, in. .485
Pitobe length, ft. 11' 55
Pitobe heater setting H

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
39	12:14		.14		8.0		108		250	50		180
40	12:16		.14		8.0		110		250	52		180
41	12:26	831.078	.02		1.15		102		255	52		160
42	12:28		.02		1.15		102		255	53		170
43	12:30		.04		2.3		103		260	54		170
44	12:32		.05		2.85		104		255	54		170
45	12:34		.08		4.6		104		250	55		170
46	12:36		.12		6.8		106		245	48		170
47	12:38		.15		8.6		108		250	48		170
48	12:40	848.064	.12		6.8		108		250	49		160
			(ΔP_{avg}) = 0.2562	~~300~~	3.987		92.4					~~160~~ 161.7
		104.388										

IMPINGER

	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER

	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

0030283

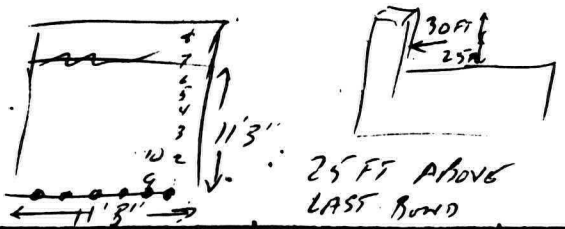
Boiler House Stack #2 Run 1 PAGE 1 OF 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant Banco - Hollows Mill
Run Number 1
Location Boiler House Stack #2
Date 9/26/79
Operator SCS
Sample case number 2
Monitor Unit number 1

Ambient temperature, °F 50
Barometric pressure, in. Hg 25.94
Assumed moisture, % 3
Heater box setting, °F ~~H~~ 3
Pitobe tip diameter, in. 0.489
Pitobe length, ft. 11 FT 5.5. (165)
Pitobe heater setting Hi
Pitot C_p = .839



$\Delta H/6 = .20$ $K = 56$

Point		2 MIN/POINT (48 POINTS) Clock Time		Dry Gas Meter ft³	$(\sqrt{\Delta P})_{avg} = 0.219$ Pitot, in. H_2O ΔP		Orifice ΔH, in. H_2O Desired	Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge		Sample Case Temperature, °F		Impinger Temperature, °F		Stack Pressure, in. Hg	Stack Temperature, °F	
1	21	START 11:06AM	12:02	806.756	0.03	0.04	1.6	2.2	67	75	6	5	225	245	40	50	25.94	160	160
2	22	08	04		0.06	0.08	3.4	4.5	68	76	6	8	225	245	40	50		160	160
3	23	10	06		0.09	0.09	5.0	5.0	69	78	8	9	255	265	40	48		160	160
4	24	12	08	811.362	0.10	0.11	5.6	6.1	70	82	9	10	260	265	46	50		160	160
5	25	14	12:14		0.10	0.035	5.6	1.9	71	80	9	5	265	225	46	47		160	160
6	26	16	20		0.09	0.03	5.0	1.65	74	80	9	5	265	225	49	48		160	160
7	27	18	22		0.11	0.02	6.2	1.1	76	80	10	5	265	225	52	48		160	160
8	28	20	24		0.09	0.03	5.0	1.65	77	80	8	5	260	225	52	45		160	160
9	29	11.30	26	825.760	0.04	0.05	2.2	2.8	74	81	5	6	225	235	41	49		160	160
10	30	32	28		0.04	0.06	2.2	3.4	74	82	5	7	225	245	41	48		160	160
11	31	34	30		0.05	0.10	2.8	5.6	74	84	5	10	225	255	41	47		160	160
12	32	36	32	8	0.06	0.10	3.3	5.6	75	85	6	10	230	250	42	49		160	160
13	33	38	12:52		0.06	0.005	3.3	0	75	83	6	0	240	230	44	42		160	160
14	34	40	54		0.06	0.01	3.3	0.55	75	87	6	3	245	230	44	42		160	160
15	35	42	56		0.09	0.02	5.0	1.1	75	82	8	5	255	250	46	42		160	160
16	36	44	12:58	842.362	0.11	0.04	6.2	2.2	75	85	10	6	235	245	46	42		160	160
17	37	11:54	1:00		0.02	0.07	1.1	3.9	74	83	4	7	260	225	44	41		150	160
18	38	56	1:02		0.05	0.10	2.8	5.6	74	86	6	9	260	225	42	42		160	160
19	39	58	1:04		0.02	0.10	1.1	5.6	75	87	4	9	260	230	42	43		160	160
20	40	12:00	1:06	884.000	0.005	0.06	0	3.4	75	87	0		260	230	42	44		160	160

IMPINGER	wt. before	wt. after	
#1	516.5	518.3	Δ = 1.8
#2	522.0	528.8	6.8
#3	430.8	432.2	1.4
#4	674.4	694.0	19.6
			29.6 GMD

FILTER	before	after	
#1	0.4085	0.4537	25.2
#2			
#3			
#4			

Δ LEAK CHECK
Pre 0.01 CFM @ 14 PSIC
Post FILTER BOX ON AT END OF TEST CFM @ PSIC

GRAIN LOAD 0.005 GR/DSCF

Maurice J. Byrne

0030284

Bradhouse Stack #2 Run 1 Page 1 of 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant ESSMCO - Hesperia Mont
Run Number 1
Location Bradhouse Stack 2
Date 5/26/79
Operator SB
Sample case number 2
Monitor Unit number 1

$V_{m\,STD}$ = 76.216 SCF
V_{s2DC} = 14.291 FT/SEC
$\%H_2O$ = 1.8%
%I50 = 98.0%

Ambient temperature, °F
Barometric pressure, in. Hg
Assumed moisture, %
Heater box setting, °F
Pitobe tip diameter, in.
Pitobe length, ft.
Pitobe heater setting

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
41	2:44		0.01	0.55		85		3	225	45	25.94	160
42	46		0.01	0.55		85		3	225	45		160
43	48		0.015	0.84		85		3	225	45		160
44	50		0.01	0.55		85		3	225	45		160
45	52		0.03	1.65		86		3	225	46		160
46	54		0.07	3.90		87		4	225	46		160
47	56		0.04	2.2		88		5	225	50		160
48	58	895.727	0.04	2.2		89		5	225	50		160
		Δ=88.971	$(\sqrt{\Delta P})_{AV}$=0.214	$\bar{x}$=3.06		$\bar{x}$=79.1						$\bar{x}$=160

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIG
Post _____ CFM @ _____ PSIG

Maurice J Byrne 6/6/79

0030285

BAG HOUSE STACK #2 RUN 2

Plant ASARCO - HELENA MONT
Run Number 2
Location BAG HOUSE STACK #2
Date 5/27/79.
Operator ScS
Sample case number 2
Monitor Unit number 1

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Vmstd = ~~76.306~~ 76.599
Vstdc = ~~14.129~~ 14.145
%H2O: ~~1.9%~~ 1.9%
%ISO = ~~98.8%~~ 99.1%
GRAIN LOAD 0.0046 gr/DSCF

Ambient temperature, °F 55
Barometric pressure, in. Hg 26.00
Assumed moisture, % 2%
Heater box setting, °F 250 (3)
Pitobe tip diameter, in. ~~0.4~~ 0.489
Pitobe length, ft. 11 FT SS (P.S.)
Pitobe heater setting Hi
Cp = ~~.839~~ .839

Point	Clock Time (2 min/point)	Dry Gas Meter ft³	Pitot, in. H_2O ΔP ($(\sqrt{\Delta P})_{AV}$ = .217)	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	11:10 AM	896.258	0.03	1.73		80		5	265	.36	26.00	150
2	12		0.06	3.5		81		6	265	36		160
3	14		0.07	4.0		82		7	265	36		160
4	16		0.09	5.1		83		9	265	37		160
5	18		0.10	5.9		84		10	265	41		160
6	20		0.11	6.4		85		10	265	45		160
7	22		0.10	5.9		86		10	270	50		160
8	24		0.08	4.6		88		10	270	52		160
9	11:32	915.481	0.04	2.3		85		5	280	44		160
10	34		0.05	3.0		85		6	280	44		160
11	36		0.05	3.0		86		6	285	43		160
12	38		0.04	2.3		87		5	285	44		160
13	40		0.05	3.0		87		6	280	46		160
14	42		0.06	3.6		88		6	280	48		160
15	44		0.11	6.4		90		10	280	48		160
16	46		0.11	6.4		91		10	275	52		160
17	11:54	932.780	0.04	2.3		88		5	265	40		160
18	56		0.04	2.3		88		5	265	40		160
19	58		0.03	1.8		88		5	265	40		160

IMPINGER	wt. before	wt. after	Δ=
#1	531.3	527.7	-3.6
#2	539.8	550.0	10.2
#3	424.5	427.5	3.0
#4	666.5	688.1	21.6
			31.2 cc H_2O

FILTER	before	after	
#19	0.4048	0.4262	21.4
#2			
#3			
#4			

LEAK CHECK
Pre <0.01 CFM @ 14 PSIG
Post <0.01 CFM @ 14 PSIG

[signature] 5/28/79

0030286

Boiler House Stack #2 Run 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant _____
Run Number _____
Location _____
Date _____
Operator _____
Sample case number _____
Monitor Unit number _____

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Probe 2013

Point	Clock Time	Dry Gas Meter ft^3	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
20	1200	~~0.00~~	0.01	0.59		88		3	265	40	26.00	160
21	02		0.06	3.5		88		6	255	38		160
22	2:16 ~~04~~		0.06	3.4		93		6	225	50		160
23	2:1~~06~~		0.09	4.9		92		8	225	45		160
24	~~08~~ ~~0~~2:20		0.10	5.6		92		9	230	47		160
25	2:30		0.02	1.2		91		4	245	45		160
26	32		0.02	1.2		91		4	245	45		~~160~~ 160
27	34		0.02	1.2		91		4	250	52		160
28	36		0.01	.56		91		3	255	52		160
29	38		0.02	1.2		92		4	250	53		160
30	40		0.07	4.0		93		7	260	53		160
31	42		0.09	5.0		93		8	260	56		160
32	44	961.685	0.09	5.0		94		9	260	58		160
33	2:58		0.02	1.1		93		4	230	58		160
34	3:00		0.00	0		93		0	230	58		160
35	02		0.01	0.56		93		3	230	60		160
36	04		0.02	1.1		93		4	235	53		160
37	06		0.06	3.4		93		6	235	52		160
38	08		0.09	4.9		94		9	230	53		160

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIG
Post _____ CFM @ _____ PSIG

Susan Ann J Byrne
EPA 6/28/79

00302877

Bag House Stack #2 Runs

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant _____
Run Number _____
Location _____
Date _____
Operator _____
Sample case number _____
Monitor Unit number _____

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Page 3 of 3

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
39	10		0.11	6.2		96		10	245	57	26.00	160
40	12	977.534	0.05	2.8		97		6	260	60		160
41	3:24		0.01	0.55		94		4	250	50		160
42	26		0.01	0.55		94		4	250	48		150
43	28		0.01	0.55		94		3	250	48		150
44	30		0.03	1.7		94		5	250	48		150
45	32		0.04	2.2		94		5	250	48		150
46	34		0.05	2.8		94		6	250	48		150
47	36		0.06	3.4		94		6	250	53		150
48	38		0.07	3.9		95		7	240	52		150
		986.946										
		90.688	$\sqrt{\Delta p}$: 0.2168	$\bar{X}$ = 3.054		$\bar{X}$ = 90.1						$\bar{X}$ = 153.3

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

0030288

Facility #2 Run 3 Page 1 of 3

Plant ASARCO - Hayden Mont
Run Number 3
Location Baghouse Stack #2
Date 9/27/79
Operator SGS
Sample case number 2
Monitor Unit number 1

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

$V_{m\,STD}$ 77.337 SCF
$V_{STOIC.}$ 14.039 Ft³/Sec
$\%H_2O$ 1.2%
% ISO ~~99.1%~~ 99.1%
Grain loading 0.005 Gr/DSCF

Ambient temperature, °F 50
Barometric pressure, in. Hg 25.92
Assumed moisture, % 2
Heater box setting, °F 250 (3.5)
Pitobe tip diameter, in. 0.489
Pitobe length, ft. 11 FT (55)
Pitobe heater setting Hi

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	8:18 AM	987.504	0.03	1.7		60	5		250	32	25.92	150
	20		0.05	~~1.7~~ 2.8		60	6		250	32		150
3	22		0.07	4.0		60	7		250	32		150
"	24		0.10	5.6		61	10		260	32		150
5	26		0.11	6.1		62	10		260	37		150
6	28		0.11	6.1		63	10		260	37		150
7	30		0.11	6.1		65	10		270	41		150
8	32		0.07	4.0		66	7		270	44		150
9	8:56	006.291	0.03	1.7		60	5		255	35		150
10	58		0.04	2.2		60	5		255	35		150
11	9:00		0.05	2.7		60	5		255	35		150
12	02		0.06	3.3		60	5		255	35		150
13	04		0.06	3.3		61	5		255	35		150
14	06		0.07	4.0		62	5		250	37		150
15	08		0.11	6.1		64	10		250	38		150
16	10		0.12	6.7		65	10		250	38		150
17	9:27	023.360	0.04	2.2		65	5		260	37		150
18	21		0.04	2.2		65	5		260	37		150
19	26		0.03	1.7		65	5		260	37		150

IMPINGER	wt. before	wt. after	
#1	522.4	517.0	-5.4
#2	541.7	552.7	11.0
#3	430.3	432.7	2.4
#4	658.0	675.5	17.5
			25.5

FILTER	before	after
#1 21	0.4090	0.4325
#2		
#3		
#4		

Δ LEAK CHECK 23.5
Pre 0.01 CFM @ 15 PSIC
Post 0.01 CFM @ 17 PSIC

Jonathan J. Byrne
EPA 9/28/79

00030289

Boiler House Stack #2 Run 3

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard

South Gate, CA 90280

Plant _____

Run Number _____

Location _____

Date _____

Operator _____

Sample case number _____

Monitor Unit number _____

Ambient temperature, °F _____

Barometric pressure, in. Hg _____

Assumed moisture, % _____

Heater box setting, °F _____

Pitobe tip diameter, in. _____

Pitobe length, ft. _____

Pitobe heater setting _____

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
20	28		0.03	1.7		66		5	260	40		150
21	30		0.03	1.7		67		5	260	41		150
22	32		0.06	3.4		68		7	265	43		150
23	34		0.10	5.7		70		10	265	44		150
24	36		0.11	6.2		71		10	270	45		150
25	9:48	038.725	0.01	0.55		70		3	260	42		150
26	50		0.01	0.55		70		3	260	42		150
27	52		0.02	1.1		70		4	260	42		150
28	54		0.03	1.7		70		5	260	41		150
29	56		0.05	2.8		71		6	230	41		150
30	58		0.05	2.8		72		6	245	42		150
31	10:00		<0.005	0		73		0	255	46		150
32	02		0.11	6.2		73		10	260	50		150
33	10:18	049.838	0.01	0.55		70		3	250	40		140 ~~150~~
34	~~20~~ 19		0.01	0.55		70		3	250	39		140 ~~150~~
35	~~22~~ 21		0.03	1.7		70		5	245	35		140
36	~~24~~ 23		0.06	3.4		70		7	245	38		140
37	~~26~~ 25		0.07	3.9		72		7	250	39		150
38	~~28~~ 27		0.07	3.9		73		7	250	44		160

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK

Pre _____ CFM @ _____ PSIC

Post _____ CFM @ _____ PSIC

Brandon J Byrne
EPA 9/29/79

0030290

Baghouse Stack #2 Run 3 Page 3 of 3

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard

South Gate, CA 90280

Plant _____
Run Number _____
Location _____
Date _____
Operator _____
Sample case number _____
Monitor Unit number _____

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Point	Clock Time	Dry Gas Meter ft^3	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
39	~~10:29~~		0.10	5.6		75		3	250	45		160
40	~~10:31~~		0.10	5.6		77		3	250	48		160
41	10:42	064.901	<0.005	~~5.6~~ 0		73		0	225	48		140
42	411		<0.005	0		73		0	225	48		140
43	46		0.01	0.55		73		3	225	44		140
44	115		0.03	1.7		73		4	225	45		140
45	50		0.06	3.4		75		6	230	44		140
46	57		0.07	3.9		76		7	240	43		150
47	511		0.07	3.9		77		7	235	46		150
48	56		0.04	2.2		77		5	250	50		140
		065.669	$\sqrt{\Delta P}$=0.2167	$\bar{X}$=3.068								
						681						x 148.6
		Δ=88.165										

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

Mark J. Byrne

00030291

BAGHOUSE STACK #2 RUN 4

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard

South Gate, CA 90280

Plant ASARCO - Helena Mont.
Run Number 4
Location Bag House Stack #2
Date 9/28/79
Operator SES
Sample case number 2
Monitor Unit number 1

$V_{m STD}$ 83.154 SCF
V_{roc} ~~15.709~~ FT/SEC 15.671
% H_2O 1.7%
% ISO 97.4%

Ambient temperature, °F 50 P1 of 3
Barometric pressure, in. Hg ~~29.94~~ 25.94
Assumed moisture, % 2
Heater box setting, °F 250
Pitobe tip diameter, in. 0.489
Pitobe length, ft. 11 FT SS. (Pos)
Pitobe heater setting Hi

Point	Clock Time (Lane/Point)	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
48	2:32 PM	076.000	0.01	0.56		94		3	260	~~260~~ 48	25.94	130
47	34		0.01	0.56		94		3	255	35		130
46	36		0.03	1.7		94		5	253	34		140
45	38		0.04	2.2		95		5	230	40		140
44	40		0.02	1.1		95		4	230	44		140
43	42		<0.005	0		95		0	230	46		140
42	44		<0.005	0		95		0	230	45		140
41	46		0.01	0.56		95		3	230	45		140
40	2:56		0.06	~~2.2~~ 3.4		95		6	260	49		~~150~~ 160
39	58		0.10	5.6		95		9	270	47		~~160~~ 150
38	3:00		0.10	5.6		96		9	275	46		150
37	02		0.06	3.4		97		6	275	47		160
36	04		0.03	1.7		97		5	255	53		160
35	06		0.04	2.2		98		5	255	56		160
34	08		0.03	1.7		99		5	255	57		160
33	10		0.03	1.7		99		5	255	50		160
32	3:20		0.11	6.2		97		10	235	46		160
31	22		0.10	5.6		98		9	235	44		160
30	24		0.11	6.2		99		10	235	44		160

IMPINGER	wt. before	wt. after	
#1	524.0	516.3	-7.7
#2	539.2	548.9	9.7
#3	428.7	432.7	4.0
#4	706.6	730.7	24.1
			30.1

FILTER	before	after	
#1 23	0.4081	0.4377	29.6
#2			
#3			
#4			

Δ LEAK CHECK
Pre <0.01 CFM @ 17 PSIC
Post <0.01 CFM @ 17 PSIC

[illegible] 0.005 [illegible]

Martin J Byrne
EPA 9/28/79

0030292

Baghouse Stack #2 Run 4 P2 of 3

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant _____
Run Number _____
Location _____
Date _____
Operator _____
Sample case number _____
Monitor Unit number _____

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
29	26	108	0.07	3.9		100		6	235	47	25.54	170
28	28		0.05	2.8		101		6	235	47		160 ~~170~~
27	30		0.04	2.2		101		5	235	47		160
26	32		0.04	2.2		101		5	235	47		160
25	34		0.04	2.2		101		5	235	47		160
24	3:48	117.093	0.10	5.6		98		9	265	50		170
23	50		0.11	6.2		98		10	265	50		170
22	52		0.08	4.5		99		7	270	49		175
21	54		0.08	4.5		99		7	270	52		175
20	56		0.08	4.5		99		7	270	52		175
19	58		0.04	2.2		100		5	265	50		150
18	4:00		0.06	3.4		100		6	265	50		150
17	02		0.08	4.5		100		7	265	50		160
16	4:29	136.319	0.12	6.7		94		10	225	46		170
15	31		0.15	8.4		94		14	225	44		170
14	33		0.11	6.2		95		10	250	47		170
13	35		0.08	4.5		97		7	250	45		170
12	37		0.05	2.8		97		6	255	45		170
11	39		0.04	2.2		96		5	260	54		175

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

Thomas J. Byrne

00302933

Baghouse Stack #2 Run 4 3 of 3

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant _____
Run Number _____
Location _____
Date _____
Operator _____
Sample case number _____
Monitor Unit number _____

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Point	Clock Time	Dry Gas Meter ft^3	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
10	41		0.04	2.2		96		5	260	54	26.94	175
9	43		0.05	2.8		96		5	260	54		175
8	5:17	155.466	0.08	4.5		85		7	270	50		160
7	19		0.11	6.2		85		10	270	50		160
6	21		0.11	6.2		86		10	270	50		160
5	23		0.11	6.2		87		10	270	49		160
4	25		0.11	6.2		89		10	270	52		175
3	27		0.09	5.0		90		9	270	54		175
2	29		0.06	3.4		90		6	270	54		180
1	31		0.04	2.2		90		5	265	55		180
		175.571	$\sqrt{\Delta P}$ = 0.2916									
		Δ: 99.591		X = 3.633		X = 96						X = 160.4

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

Marilyn J. Byrne
CTL Ohio

0030294

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant ASARCO - Hayden Mont
Run Number 1
Location ~~Inlet Hood~~ Baghouse Final Stack 3
Date 9/26/79
Operator RJD
Sample case number 1
Monitor Unit number 2

Ambient temperature, °F ~~70~~ 55
Barometric pressure, in. Hg ~~25.84~~ 25.94
Assumed moisture, % 5
Heater box setting, °F
Pitobe tip diameter, in. .485
Pitobe length, ft. 11 FT 5.5
Pitobe heater setting #1

Cp = .819
$V_{m\,std}$ ~~65,165~~ 65,192 scf
Velocity ~~12,724~~ 12,746 ft/sec
%H_2O ~~2.0~~ 2.0%
% I 50 ~~98.2~~ 98.2%

2 min each.

K-55 ~~K-53~~

Point	Clock Time	Dry Gas Meter ft³	$(\sqrt{\Delta p})_{AV}$ = 0.197 Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
			.02									
1	11:06	396.510	~~.03~~	~~1.65~~	1.10		66	4 ~~5~~			25.~~84~~	160°
2			.05		2.75		66	5	230	~~70~~ 60		160
3	11:10		.06		3.30		72	5	230	49		
4	11:12	403	.07		3.85		74	6	235	51		160
5	11:14		.07		3.85		76		220	54		160
6	11:16	405	.09		4.40		78	7	230			
7	11:18	~~.008~~	.08		4.40		78	7	230	58		160
8	11:20	412.411	.08		4.40		80	7		58		170
9	11.30	412.411	.03		1.65		80	4	230			150
10	11:32		.03		1.65					50		
11	11:34		.03		1.65		80		220	48		150
12	11:36		.04		2.20		82	5	"	"		160
13	11:38		.04		2.20		84	5	"	"		160
14	11.40		.05		2.75		84	5	230	49		160
15	11:42		.07		3.85		84	5	"	50		180
16	11:44		.07		3.85		84	"	"	"		190
17	11:54	426.259	.02		1.10		84	3	235			180
18	11:56		.04		2.20		84	5	24~~0~~5	52		185
19	11:58		.03		1.65		86		260	50		180

IMPINGER	wt. before	wt. after	Δ
#1	541.4	547.1	5.7
#2	539.1	544.3	5.2
#3	429.6	431.4	1.8
#4	624.8	639.7	14.9
			27.66 H_2O

#0017

FILTER	before	after
#1	.4038	0.4222
#2		
#3		
#4		

Δ LEAK CHECK 11/16
184 Pre <0.01 CFM @ 15 ~~PSIG~~
Post <0.01 CFM @ 15 ~~PSIG~~

Grain loading 0.004 gr/dscf

Maurice J. Dyer
CTA

0030295

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant Asarco - Helena Mt.
Run Number 1
Location Baghouse #3
Date 7-26-79
Operator RDD
Sample case number 1
Monitor Unit number 2

Ambient temperature, °F 55
Barometric pressure, in. Hg 25.94
Assumed moisture, % 5
Heater box setting, °F 240°
Pitobe tip diameter, in. .485
Pitobe length, ft. 11 ft 55
Pitobe heater setting Hi

r_{CO_2} = 0.8%
O_2 = 20.2%
CO = 0.2%
M_{wDry} = 28.94

page 2

PORT	Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
	20	12:00		.03		1.65		88	4	260	50		180
3	21	12:02		.02		~~1.10~~			"	240	50		180
	22	12:04		.02		1.10		88	"	235	50		180
	23	12:06		.07		3.85		88		235	52		180
	24	12:08		.09		4.95		90	"	235	52		180
	25	12:14	438.615	.02		1.10		86		240	52		180
	26	12:20		.03		1.65		86	"		~~55~~		
	27	12:22		.03		1.65		88		240	95		190
4	28	12:24		.02		1.10		86	"	240	57		200
	29	12:26		.03		1.65		86		240	57		200
	30	12:28		.04		2.20		88		240	58		200
	31	12:30		.06		3.30		90		245	58		
	32	12:32		.06		3.30		90		245	58		190
	33	12:52	450.774	.01		0.55		82		250	52		180
	34	12:54		.01		0.55		82		240	52		180
5	35	12:56	453.842	.02		1.10		84		235	54		180
	36	2:24		.03		1.65		82		255	58		180
	37	2:26		.04		2.20		84	5	255	56		
	38	2:28		.05		2.75		84		250	54		

IMPINGER	wt. before	wt. after
#1	See Sheet #1	
#2		
#3		
#4		

FILTER	before	after
#1	See Sheet #1	
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIG
Post _____ CFM @ _____ PSIG

Martin J. Cyrus
EPA 936-74

00030296

Plant ASARCO - Helena Mt.
Run Number 1
Location Baghouse #3
Date 9-26-79
Operator RJR
Sample case number 1
Monitor Unit number 2
$V_{m\,std}$ 65.465
V_{aqc}.
$\%H_2O$
$\%CO_2$

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

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Ambient temperature, °F 60
Barometric pressure, in. Hg 25.94
Assumed moisture, % 5-%
Heater box setting, °F 250
Pitobe tip diameter, in. .485
Pitobe length, ft. 11' 5S
Pitobe heater setting Hi

PORT	Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F	Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
5	39	2:30		.06		3.30	86		260	58		180
	40	2:32		.06		3.30	88		260	58		180
	41	2:44	463.998	<.01		0	88		250	58		180
	42	2:46		.01		.55	88		250	59		180
	43	2:48		.01		.55	88		250	60		180
6	44	2:50		.02		1.10	88		260	56		180
	45	2:52		.05		2.75	90		245	50		
	46	2:54		.06		3.30	92		230	52		180
	47	2:56		.07		3.85	94		230	52		180
	48	2:58		.07		3.85	96		255	54		180
	STOP		473.608									
			Δ=77.298	$\left(\frac{\Delta P}{m}\right)^2$=0.197		$\bar{x}$=2.349	$\bar{x}$=89.2					$\bar{x}$=176.4

IMPINGER — wt. before / wt. after
#1
#2
#3
#4

FILTER — before / after
#1
#2
#3
#4

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

0030297

Bag House Stack #3 Run 2

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant ASARCO Helena
Run Number 2
Location Bag house #3
Date 9-27-79
Operator Bob
Sample case number 1
Monitor Unit number 2

$V_{m\,std}$ ~~67.557~~ SCF 67.275
$V_{s\,actual}$ 12.638 FT/SEC 12.638
%H_2O 1.9%
% ISO ~~101.2%~~ 100.7%

Ambient temperature, °F 55
Barometric pressure, in. Hg 26.00
Assumed moisture, % 2%
Heater box setting, °F 250
Pitobe tip diameter, in. 0.485
Pitobe length, ft. 11' 55
Pitobe heater setting Hi
C_p = .819
K = 57

2 min

6 min 10 sec 0.003 GAL/DSCF

Point	2 min/Point 48 Points Clock Time	Dry Gas Meter ft³	$(\Delta P)_{Av}$ = 0.1968 Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	11:10	474.479	.03		1.71		74		230	.50		160
2	11:12		.04		2.3		76		240	44		160
3	11:14		.05		2.85		78		240	44		160
4	11:16		.07		4.0		80		235	46		160
5	11:18		.08		4.6		82					
6	11:20		.08		4.6		86		245	46		160
7	11:22		.08		4.6		88	6"	235	54		180
8	11:24		.08		4.6		90		240	54		
9	11:32	490.902	.03		1.7		88		240	52		180
10	11:34		.04		2.3		90		240	46		180
11	11:36		.04		2.3		92		250	46		180
12	11:38		.04		2.3		94		255	44		170
13	11:40		.05		2.85		96		250	44		180
14	11:42		.06		3.4		98	6"	240	46		180
15	11:44		.08		4.6		100		250	50		
16	11:46		.09		5.1		104	7"	250	52		180
17	11:54	506.315	.02		1.14		98		240	48		180
18	11:56		.05		2.85		98		245	46		180
19	11:58		.02		1.14		98		240	50		

IMPINGER	wt. before	wt. after	Δ
#1	539.3	543.4	4.1
#2	538.6	545.0	6.4
#3	429.2	431.1	1.9
#4	679.7	695.5	15.8

Impinger Soln 0.1N HNO_3

28.2 cc H_2O

0016

FILTER	before	after
#1	.4092	0.4243
#2		
#3		
#4		

Δ LEAK CHECK 15.1 mc
Pre <0.01 CFM @ 15 PSIG
Post <0.01 CFM @ 15 PSIG

Brenda J. Byrne
9/27/79

0030298

Plant Asarco - Helena, Mt.
Run Number 2
Location Bag House #3
Date
Operator
Sample case number
Monitor Unit number

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

page 2

Ambient temperature, °F
Barometric pressure, in. Hg
Assumed moisture, %
Heater box setting, °F
Pitobe tip diameter, in. .485
Pitobe length, ft. 11' 5S
Pitobe heater setting H

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
20	12:00		.03		1.71		98	3"	255	52		180
21	12:02		.03		1.71		98		255	50		
22	12:04	513.373	.05		~~2.85~~ 2.85		82		225			160
23	2:15		.05		2.85		82					
24	2:17		.07		4.0		82	"	235	52		160
25	2:30	519.399	.02		1.15		84	"	235	48		160
26	2:32		.02		1.15		85		235	48		160
27	2:34		.02		1.16		86		240	50		160
28	2:36		.02		"		"		"	"		
29	2:38		.03		1.71		87		250	52		160
30	2:40		.04		2.3		88		255	54		160
31	2:42		.05		2.85		90		245	59		
32	2:44		.05		2.85		92		245	61		160
33	2:58	531.542	.01		0.55		88		240	58		160
34	3:00		.02		1.15		88		235	52		160
35	3:02		.02		1.15		89		250	54		
36	3:04		.03		1.71		90		255	54		160
37	3:06		.03		1.71		90		245	56		160
38	3:08		04		2.3		92		250	54		

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

Brandon P Byrne
EPA 6/26/74

0030299

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant Asarco - Helena
Run Number 2
Location Baghouse #3
Date 9-27-79
Operator PGV
Sample case number
Monitor Unit number

page 3

Ambient temperature, °F
Barometric pressure, in. Hg
Assumed moisture, %
Heater box setting, °F
Pitobe tip diameter, in.
Pitobe length, ft.
Pitobe heater setting

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
39	3:10		.06		3.4		94		250	.54		170
40	3:12		.07		4.0		94	5"	"	"		"
3:24 41	3:14	544.294	<0.005		0							
42	3:26		.005		.27		92		245	56		170
43	3:28		.02		1.15		92		245	51		170
44	3:30		.02		1.15		92		240	59		170
45	3:32		.03		1.71		92		240	54		180
46	3:34		.05		2.85		93					180
47	3:36		.06		3.4		95		240	46		170
48	3:38	554.896	.06		3.4		96		"	"		"
		Δ=80.417	$(\Delta p)_{Ar}$=0.1968		$\bar{x}$=2.42		$\bar{T}$=90.0					$\bar{x}$=168.3

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

0030300

Baghouse Stack #3 Run 5 Page 1 of 3

Plant Banco - Horizontal Mono
Run Number 5
Location Baghouse Stack #3
Date 9/29/79
Operator SS
Sample case number 2
Monitor Unit number 1

CTL - ENVIRONMENTAL SERVICES
2905 E. Century Boulevard
South Gate, CA 90280

$V_{m\ STD}$ ~~72.8475~~ SCF 72.787
$V_{w\ REC.}$ 13.907 FT³
$\%H_2O$ 1.9%
%ISO 98.4%
GRAIN LOAD 0.0036 GR/DSCF

Ambient temperature, °F 50
Barometric pressure, in. Hg 26.07
Assumed moisture, % 2
Heater box setting, °F 250 (4)
Pitobe tip diameter, in. 0.489
Pitobe length, ft. 11FT (SS.) Abs
Pitobe heater setting Hi
C_p = .839
METER CORR FACTOR = 1.004

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
1	10:32 AM	175.918	0.04	2.2		60		6	275	33	26.07	140
2	34		0.06	3.4		61		7	275	33		140
3	36		0.07	3.9		62		8	265	33		140
4	38		0.08	4.5		64		9	265	33		140
5	40		0.09	5.0		65		10	260	36		140
6	42		0.10	5.6		68		11	260	40		140
7	44		0.09	5.0		70		10	260	41		140
8	46		0.06	3.4		71		7	260	35		140
9	10:54	194.004	0.04	2.2		69		6	260	42		140
10	56		0.03	1.7		70		5	260	42		140
11	58		0.04	2.2		70		5	250	42		140
12	11:00		0.03	1.7		71		4	255	42		140
13	02		0.045	2.5		72		6	260	41		140
14	04		0.06	3.4		72		7	255	42		140
15	06		0.09	5.0		74		10	260	42		140
16	08		0.10	5.6		75		11	260	45		140
17	11:16	209.272	0.04	2.3		73		6	270	41		140
18	18		0.03	1.7		74		5	270	41		140
19	20		0.03	1.7		74		5	270	41		140

IMPINGER	wt. before	wt. after	
#1	539.4	534.9	-4.5
#2	538.6	551.2	12.6
#3	424.4	427.9	3.5
#4	694.3	713.2	18.9
			30.5

FILTER	before	after	Δ
#1 25	0.4080	0.4230	15.0
#2			
#3			
#4			

Δ LEAK CHECK
Pre <0.01 CFM @ 17 PSIC
Post <0.01 CFM @ 17 PSIC

0030301

Boilerhouse Stack #3 Run 5

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard

South Gate, CA 90280

Plant _____
Run Number _____
Location _____
Date _____
Operator _____
Sample case number _____
Monitor Unit number _____

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O		Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
				Desired	Actual							
20	22		0.02	1.1		74		4	265	41		140
21	24		0.03	1.7		74		5	270	41		140
22	26		0.04	2.2		74		6	270	41		140
23	28		0.07	3.9		75		8	265	42		140
24	30		0.10	5.6		76		11	260	42		140
25	11:40	223.339	0.02	1.1		75		4	260	42		140
26	42		0.02	1.1		75		4	265	42		140
27	44		0.01	0.56		75		3	260	42		140
28	46		0.02	1.1		75		4	255	43		140
29	48		0.03	1.7		75		5	255	41		140
30	50		0.05	2.8		75		6	260	40		140
31	52		0.06	3.4		75		7	255	41		140
32	54		0.09	5.1		76		10	255	43		140
33	12:02	235.943	0.02	1.1		75		4	260	48		140
34	04		0	0		75		0	260	48		140
35	06		0.01	0.56		75		3	260	50		140
36	08		0.03	1.7		75		5	265	45		140
37	10		0.05	2.8		75		6	260	44		135
38	12		0.05	2.8		76		6	260	44		135

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

0030302

Baghouse Stack #3 Run 5
Page 3 of 3

CTL - ENVIRONMENTAL SERVICES

2905 E. Century Boulevard
South Gate, CA 90280

Plant _____
Run Number _____
Location _____
Date _____
Operator _____
Sample case number _____
Monitor Unit number _____

Ambient temperature, °F _____
Barometric pressure, in. Hg _____
Assumed moisture, % _____
Heater box setting, °F _____
Pitobe tip diameter, in. _____
Pitobe length, ft. _____
Pitobe heater setting _____

Point	Clock Time	Dry Gas Meter ft³	Pitot, in. H_2O ΔP	Orifice ΔH, in. H_2O Desired	Orifice ΔH, in. H_2O Actual	Dry gas temperature, °F		Pump Vacuum in. Hg Gauge	Sample Case Temperature, °F	Impinger Temperature, °F	Stack Pressure, in. Hg	Stack Temperature, °F
35	14		0.07	3.9		77		7	255	45		140
40	16		0.09	5.1		79		10	255	45		140
41	12:26	248.261	0.0	0		75		0	250	60		140
42	28		0.0	0		75		0	250	50		140
43	30		0.01	0.56		75		3	250	50		140
44	32		0.02	1.1		75		4	250	49		140
45	34	0.05	~~0.02~~	~~1.1~~ 2.8		75		6	245	44		140
46	36		0.05	2.8		75		6	240	44		140
47	38		0.08	4.5		76		9	245	45		140
48	40		0.10	5.6		77		11	230	47		140
		259.259										
		Δ=85.341	$(\sqrt{\Delta P})_{Avg}$ = 0.2041			X=72.9						X=140

IMPINGER	wt. before	wt. after
#1		
#2		
#3		
#4		

FILTER	before	after
#1		
#2		
#3		
#4		

LEAK CHECK
Pre _____ CFM @ _____ PSIC
Post _____ CFM @ _____ PSIC

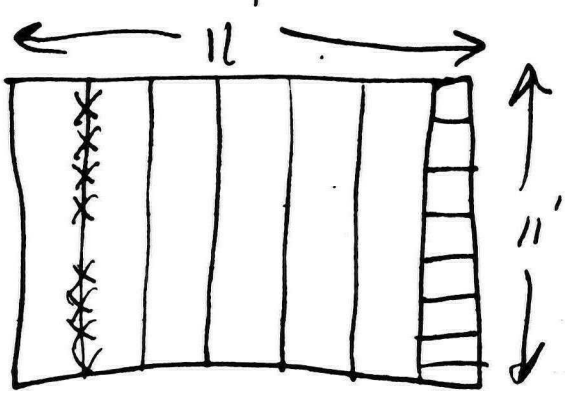
0030303

APPENDIX G

BAGHOUSE STACK SAMPLING POINTS

equiv diameter = 11

0030305



132"

for 6x8 sample pond

Point #		% to	Return	Distance in	
1	A	3.3	8:14	4.4	
2	B	10.5	+16.5	13.9	1' 1.9"
3	C	19.4	+16.5	25.6	2' 1.6"
4	D	32.3	+16.5	42.4	3' 6.6"
5	E	67.7	+16.5	89.4	7' 5.4"
6	F	80.6	+16.5	106.4	8' 10.4"
7	G	89.5	+16.5	118.1	9' 10.1"
8	H	96.7	+8:14	127.6	10' 7.6"

Nozzle diameter measurement ~.250

Point	Diam In A	B
1	.251	.252
2	.251	.251
3	.255	.250
4	.253	.249
5	.254	.251
6	.253	.250
7	.252	.249
8	.251	.249
9	.254	.248
10	.253	.250
Ave	.253	.250

APPENDIX H

STACK TEST EQUIPMENT CALIBRATION

9/25/79 STACK TEST EQUIP. CALIBRATION

CONTROL UNIT #1 (ΔH@ = 2.27)

CALIB. AGAINST ROCKWELL DRY TEST METER #SN 351477 MODEL T-110.

ROCKWELL CORRECTION DATA:

CFM	% ERROR	
.33	0.0%	CALIBRATED BY ROCKWELL
.66	+0.3%	12/27/76
1.00	+0.21%	(CURVE ON FILE WITH
1.33	0.0%	METER)
1.66	0.0%	

* = CORRECT AS ABOVE

UNIT #1

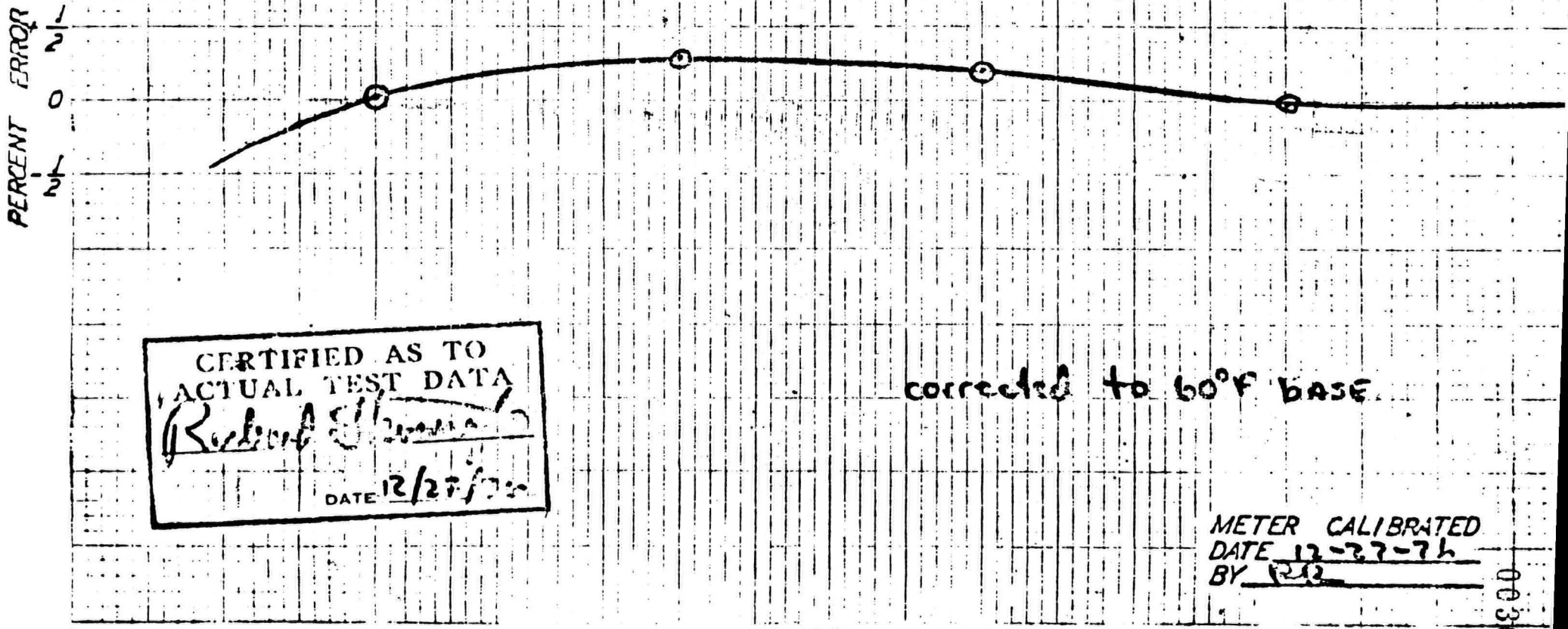
ΔH (INCHES)	TEST METER	UNIT	SCFM	γ	ΔH@
0.5	178.842	791.033	0.360 SCFM		(2.17)
	186.201	798.710			
	7.359	7.677			
	7.350* CF				
	6.501 SCF	6.488 SCF		γ = 6.501/6.488 = 1.002	
1.0	164.151	775.772			
	171.402	783.325	0.497 SCFM		(ΔH@ 2.27)
	7.251 CF	7.553			
	7.237* CF				
	6.401 SCF	6.392 SCF		γ = 6.401/6.392 = 1.001	

Rockwell International

DUBOIS, PA

TEST METER PERFORMANCE
METER SIZE T-10
S/N 351023
CUSTOMER T.C. Ball Co.
Los Angeles, Ca.

CURVE NO. 1



CERTIFIED AS TO
ACTUAL TEST DATA
Robert Gilmore
DATE 12/27/74

corrected to 60°F base.

METER CALIBRATED
DATE 12-27-74
BY RG

00030308

$\Delta H_@ = 2.29$

0030309

CONTROL UNIT #2 CALIBRATION

ΔH	TEST	UNIT #2	SCFM	γ
0.5	198.058	373.919		
	201.269	377.302	0.353 SCFM	
	3.211 CF	3.383		
	3.207*			
	2.837 SCF	2.849 SCF	$\gamma = \frac{2.837}{2.849} = 0.996$	

$\Delta H_@ = 2.34$

1.0	201.343	377.384		
	205.215	381.491		
	3.872 CF	4.107	0.495 SCFM	
	3.864* CF			
	3.418 SCF	3.444 SCF	$\gamma = \frac{3.418}{3.444} = 0.992$	

$T_{m\ START} = 56°F$ $T_{m\ STOP} = 60°F$ $T_{AVG} = 58°F$

2.0 INCHES	207.117	383.384	
	211.589	387.812	
	4.472 CF.	4.428	
	4.459*		
	3.944 SCF	3.954 SCF	$\gamma = \frac{3.944}{3.954} = 0.997$

$T_{START} - 60$ T_{STOP} $T_{AVG} 66$

4.0	212.040	388.254	
	220.217	396.365	
	8.177 CF	8.111	
	8.157* CF		

7.716

00030310

2.0 INCHES

171.682 783.619

178.663 790.847

6.981 CF 7.228 CF 0.698 SCFM

6.962 CF*

(23)

6.158 SCF 6.135 SCF $\gamma = \frac{6.158}{6.135} = 1.004$

4.0 INCHES

186.521 799.043

193.752 806.467

7.231 CF 7.424 CF

7.247* 0.998 SCFM

(Note 2.34)

6.383 SCF 6.336 SCF $\gamma = \frac{6.383}{6.336} = 1.007$

AVERAGE $\gamma = 1.004$

OH

0.5

1.0

2.0 INCHES

4.0

APPENDIX I

PITOT TUBE CALIBRATION

9-20-79 Asarco

Cp Calculation

measured simultaneously

	Pitot type	Measured ΔP		Cp
(1)	5' S Type CTL	.98	.98	> .825
	Standard Pitot	.68	.68	
(2)	11' S Type CTL	.98	.98	> .819
	Standard	.67	.67	
(3)	11' S Type PES	.86	.87	> .838
	Standard	.62	.62	

Nozzle Diameter measurement ~ 0.5"

Run	A	B
1	.485	.482
2	.488	.486
3	.490	.487
4	.491	.483
5	.490	.485
6	.489	.486
7	.490	.483
8	.488	.487
9	.489	.484
10	.490	.485
Ave	.489	.485